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Deinking method and deinking composition.

To provide a deinking agent having an excellent ability to remove ink and a good workability whereby a deinked pulp having a high b value and contaminated with little unliberated ink can be obtained either by the flotation method, the washing method or a compromise procedure thereof.

A deinking agent which 1) comprises a mixture containing higher fatty acid(s) having 8 to 24 carbon atoms or salt(s) thereof, wherein the average carbon atom number of fatty acids in the mixture ranges from 12.7 to 22.5, the content of higher fatty acid(s) having 20 to 24 carbon atoms or salt(s) thereof ranges from 9.6 to 70.6% by weight and the iodine value (IV) is not more than 45, and 2) does not include alkylbenzenesulfonates.

The deinking agent may further comprise an alkylene oxide adduct to an alcohol, a carboxylic acid or a mixture of natural oil and polyhydric alcohol or a sulfonate of the adduct.

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This invention relates to a deinking agent and a deinking method to be used for the reclamation of waste papers including newspapers, magazines and office automation (OA) waste papers. More particularly, it relates to a deinking agent whereby a deinked pulp having a high b value and contaminated with little unliberated ink can be obtained by deinking, for example, newspapers, magazines or OA waste papers through the flotation method, the washing method or a compromise procedure thereof.

It has been a practice to reclaim waste papers including newspapers, magazines and OA waste papers. Recently the effective utilization of waste papers has become more and more important in conjunction with the problems of the earth environment such as the conservation of forest resources and the refuse disposal. Further, it has been attempted to apply deinked pulp to a purpose of a higher rank, for example, from old newspapers to paper of moderate grade. On the other hand, recent improvements in printing techniques, printing systems and printing ink compositions have made it difficult to deink waste papers. In order to facilitate deinking, therefore, attempts have been made to improve deinking devices. In order to remove inks and other impurities from waste paper, there have been used alkaline agents such as caustic soda, sodium silicate, sodium carbonate and sodium phosphate, bleaching agents such as hydrogen peroxide, hydrosulfites and hypochlorites and sequestering agents such as EDTA and DTPA together with deinking agents including anionic surfactants such as alkylbenzenesulfonates, higher alcohol sulfates, a-olefinsulfonates and dialkyl sulfosuccinates, ethylene oxide adducts of higher alcohols, alkylphenols and fatty acids and nonionic surfactants such as alkanolamides, either alone or in the form of a mixture thereof. Although these deinking agents show excellent foaming properties in the flotation treatment, their abilities to collect ink are limited. In the washing method, on the other hand, they are poor in detergency and, furthermore, the good foaming properties thereof cause troubles in draining. As a result, only a deinked pulp of a low grade can be obtained thereby. Even though a pulp of a high whiteness is obtained, the dark color restricts the utilization of the deinked pulp (for example, employed in a decreased amount under the surface of cardboard or added in a decreased amount to newspapers). Alternately it is unavoidable to elevate the amount of a bleaching agent so as to do away with the darkness. In order to obtain a deinked pulp of a not dark but light color tone, it is required to elevate the b value of the Lab color space of Hunter's color difference formula. A deinked pulp of a high b value means that fine ink spots of 4 µm or below have been removed at a high ratio. The color tone of a pulp is lightened with an increase in b value. As a result, it becomes possible to lower the content of a bleaching agent such as hydrogen peroxide, to use the deinked pulp at a high ratio and to apply the deinked pulp to a purpose of a higher grade.

There are two methods for elevating the b value. One of them comprises efficiently removing the fine ink spots of 4  $\mu$ m or below, while the other comprises using a large amount of an alkali. However the latter method suffers from some disadvantages including increases in the sticky matters, the drainage load and the brittleness of the pulp. Although there have been reported some techniques regarding the former method, i.e., collecting and removing the fine ink spots of 4  $\mu$ m or below, none of them can give a satisfactory effect.

Fatty acids have been known for a long time as deinking agents of a high ability to collect ink. For example, Japanese Patent No. 80988, Japanese Patent No. 82089 and Japanese Patent No. 83901 each disclose the use of a fatty acid (in form of a soap) as a deinking agent of a high ability to collect ink.

However, the use of known fatty acids or salt(s) thereof is disadvantageous in that a large amount of ink remains unliberated.

Recently, a deinking agent of a high performance comprising a compound, to which an alkylene oxide such as ethylene oxide or propylene oxide has been added, and capable of substantially improving the whiteness of a deinked pulp has been invented (Japanese Patent Laid-Open No. 109696/1983 and Japanese Patent Laid-Open No. 130400/1984.

However it is impossible to obtain a deinked pulp of a high b value by using any deinking agent disclosed in the above literature.

U.S. Patent No. 4,231,841 discloses a deinking agent which includes five essential components. Example 1 of the specification discloses a deinking agent composition which consists of (A) 21% by weight of a sodium salt of higher fatty acid, (B) 17% by weight of a nonionic surfactant, (C) 5% by weight of a sodium linear alkylbenzenesulfonate, (D) 3% by weight of sodium carboxymethylcellulose and (E) 54% by weight of sodium metasilicate.

However, when a higher fatty acid is combined with an alkylbenzenesulfonate which is an essential component of the above prior art, brightness of the deinked paper is reduced remarkably due to the dispersibility of the alkylbenzenesulfonate, and both the b value of the paper and the retention of pulp go down. These deteriorations are more remarkable when used water (white water) is recycled in actual field.

The present inventors have conducted extensive studies in order to develop a deinking agent and deinking method which shows a good deinking performance and a good workability in the flotation method,

washing method or a compromise procedure thereof and thus gives a deinked pulp having, in particular, a high b value and contaminated with little unliberated ink.

As a result, they have surprisingly found out that these problems can be solved by using a deinking agent which 1) comprises a mixture (b) including higher fatty acid(s) having 8 to 24 carbon atoms or salt(s) thereof, wherein the average carbon atom number of the fatty acid(s) or salt(s) thereof ranges from 12.7 to 22.5, the content of higher fatty acid(s) having 20 to 24 carbon atoms or salt(s) thereof ranges from 9.6 to 70.6% by weight and the iodine value (IV) is not more than 45, and 2) does not include alkylbenzenesul-fonates.

Accordingly, the present invention provides a deinking method which comprises adding a deinking agent which 1) comprises a mixture (b) containing higher fatty acid(s) having 8 to 24 carbon atoms or salt(s) thereof, wherein the average carbon atom number of the fatty acids in the mixture ranges from 12.7 to 22.5, the content of higher fatty acid(s) having 20 to 24 carbon atoms or salt(s) thereof ranges from 9.6 to 70.6% by weight and the iodine value (IV) is not more than 45, and 2) does not include alkylbenzenesulfonates in a process for the reclamation of waste papers.

The present invention also provides a deinking agent which comprises the above mixture (b), does not include alkylbenzenesulfonates and which comprises at least one surfactant (a) selected from the group consisting of compounds represented by the following general formulae (a-1) to (a-3) and a reaction product (a-4) obtained by adding an alkylene oxide to a mixture of a natural fat with a polyhydric alcohol:

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wherein R<sub>1</sub> represents an alkyl or alkenyl group having 8 to 24 carbon atoms or an alkylphenyl group having an alkyl group carrying 6 to 14 carbon atoms;

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain when two or more kinds of the alkylene oxide are present; and p is such a number of 1 or above as to give the total molecular weight of from 800 to 10,000;

$$R_2$$
-COO-(-AO-)<sub>m</sub>-H (a-2)

wherein R<sub>2</sub> represents an alkyl or alkenyl group having 7 to 23 carbon atoms; AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain when two or more kinds of the alkylene oxide are present; and m is such a number of 1 or above as to give the total molecular weight of from 800 to 10,000; and

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F_3 - O_{-}(-AO_{-})_n - SO_3 M (a-3)
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wherein R<sub>3</sub> represents an alkyl, alkenyl or cycloalkyl group having 8 to 24 carbon atoms or an alkylphenyl group having an alkyl group carrying 8 to 12 carbon atoms;

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain when two or more kinds of the alkylene oxide are present;

n ranges from 1 to 5; and M represents an alkali metal or ammonium.

In the deinking agent wherein the components (a) and (b) are used, it is preferable that the weight ratios of the components (a-1) to (a-4) to the component (b) each falls within the following range:

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(a-1)/(b) = 10/90 - 90/10;

(a-2)/(b) = 10/90 - 90/10;

(a-3)/(b) = 5/95 - 30/70; and

(a-4)/(b) = 10/90 - 70/30.
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The present invention moreover provides a deinking method which comprises adding the above deinking agent comprising the components (b) and (a), but not including alkylbenzenesulfonates, in a process for the reclamation of waste papers.

In this method, the component (a) may be added exclusively in a stage of disintegrating (pulping) waste papers while the component (b) may be added exclusively in a mixing stage and/or a flotation stage following the pulping stage.

Since the numerical values specified with respect to the fatty acid mixture according to the present invention are critical ones, any compound similar thereto can never exert the remarkable effects of the

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present invention unless it satisfies the specification of the present invention. Therefore the specification of the numerical values (for example, the carbon atom number of the compound) in the present invention is very important. As will be clearly shown in the Examples and Comparative Examples given hereinafter, a mixture of an average carbon atom number smaller than 12.7 has a poor effect of aggregating ink, which makes it impossible to obtain a deinked pulp of a high b value. When the average carbon atom number exceeds 22.5, on the other hand, the insufficient foaming properties in the flotation stage makes it difficult to remove the aggregated ink from the system. When the content of the fatty acids having 20 to 24 carbon atoms is smaller than 9.6% by weight, the effect of aggregating fine ink spots is deteriorated and thus any deinked pulp of a high b value cannot be obtained. When the content of said fatty acids exceeds 70.6% by weight, on the other hand the deinking ability of the agent is weakened. As a result, the obtained pulp contains a large amount of unliberated ink and thus has a poor appearance. In the deinking agent of the present invention, fatty acids having 8 to 24 carbon atoms can be arbitrarily blended within the range as specified in the present invention. It is particularly preferable to contain 2.0 to 33.2% by weight of a fatty acid having 20 carbon atoms or a salt thereof and 9.5 to 32.0% by weight of a fatty acid having 22 carton atoms or a salt thereof.

When the iodine value (IV) of the deinking agent exceeds 45, only insufficient foaming properties are achieved in the flotation stage and thus the aggregated ink on the foam layer can be hardly removed from the system. As a result, the obtained deinked pulp has a low whiteness. In this case, furthermore, a large amount of unliberated ink remains in the pulp due to the poor ability to liberate ink.

As described above, the deinking agent of the present invention may arbitrarily contain fatty acid(s) having 8 to 24 carbon atoms or salt(s) thereof within the range as specified in the present invention. Particular examples of these materials include caprylic acid, pelargonic acid, capric acid, undecanoic acid, lauric acid, tridecanoic acid, myristic acid, pentadecanoic acid, palmitic acid, margaric acid, stearic acid, oleic acid, elaidic acid, linoleic acid, linolenic acid, stearolic acid, ricinolic acid, ricinelaidic acid, non-adecanoic acid, arachidic acid, heneicosanoic acid, behenic acid, brassidic acid, erucic acid, tricosanoic acid, tetracosanoic acid, coconut oil fatty acids, beef tallow fatty acids, palm oil fatty acids, tall oil fatty acids, rapeseed oil fatty acids, fish oil fatty acids, those obtained by semi-hardening or hardening these fatty acids and salts of all of these fatty acids. Examples of the salts include sodium, potassium, ammonium, magnesium and calcium salts. Among these materials, semi-hardened or hardened fish oil fatty acids or salts thereof are preferable from the viewpoints of cost and workability (i.e., usable alone). Examples of the fish oil, from which the fish oil fatty acids to be used in the present invention are obtained, include cod oil, sardine oil, saury oil, mackerel oil, herring oil, menhaden oil and those collected during the refining process of these fish oils.

Most of the fatty acids to be used as a constituent of the deinking agent of the present invention originate from natural fats. Therefore these fatty acids may be produced by conventionally known methods, for example, Twitchell decomposition, moderate-pressure catalytic decomposition or high-pressure continuous decomposition. The iodine value (IV) may be usually lowered by hydrogenation with the use of a nickel catalyst at a high temperature under elevated pressure.

As described above, though the deinking agent of the present invention exerts an excellent deinking performance when it is used alone, the combination with other surfactants is not excluded as long as these other surfactants do not impair the effect of the present invention.

For this reason the deinking agent of the present invention does not include alkylbenzenesulfonates which impede the performance of the invention due to strong dispersibility.

An unexpected and superior deinking performance of the deinking agent of the present invention was found in comparison to an agent used in combination with alkylbenzenesulfonates.

The deinking agent of the present invention may be added in any stage to thereby give a deinked pulp of improved qualities. It may be generally added in the mixing or flotation stage. Alternately, it may be added in both of these stages. When water of a particularly high hardness is used, it is preferable to add the deinking agent immediately before the flotation stage. When it is to be used in portions in each stage, it may be added in the pulping, kneading, dispersing, chemical mixing and refining stages. The ratio (by weight) of the amount of the deinking agent to be added in the former stages to that to be added in the latter ones may preferably ranges from 10/90 to 90/10, still preferably from 40/60 to 60/40. The deinking agent may be preferably added in such an amount that the workability is not deteriorated and the procedure may be effected economically. It may be preferably used in an amount of from 0.03 to 1.0% by weight based on the waste papers.

Although the working mechanism of the deinking agent of the present invention has not been clarified in detail, it is assumed to proceed as follows.

When the carbon atom number of a higher fatty acid is elevated, the adsorption of the deinking agent is

oriented almost perpendicularly to the surface of fine ink spots. As a result, the density of the terminal functional groups of the deinking agent is lowered. Thus the absolute surface charge density thereof per unit area is lowered, which might promote the aggregation of the fine ink spots, in accordance with DLVO theory. When the content of higher fatty acids having 20 to 24 carbon atoms is lower than 9.6% by weight, the fine ink spots would hardly aggregate. When this content exceeds 70.6% by weight, on the other hand, a rapid decrease in the adsorption ratio of the deinking agent onto the ink surface would make the control of the ink-surface charge density by the deinking agent insufficient. As a result, the fine ink spots would hardly aggregate.

When the content of the higher fatty acids having 20 to 24 carbon atoms ranges from 9.6 to 70.6% by weight, therefore, the fine ink spots are aggregated and thus a deinked pulp of a high b value can be obtained.

The unliberated ink can be reduced by lowering the surface tension between the ink and cellulose. When the content of the higher fatty acids having 20 to 24 carbon atoms exceeds 70.6% by weight, the critical micelle concentration rapidly increases. In this case, therefore, the performance of the deinking agent is exerted below the critical micelle concentration during the practical deinking process. Thus any satisfactory deinking effect cannot be achieved.

As described above, the content of the higher fatty acids having 20 to 24 carbon atoms or salt(s) thereof should critically range from 9.6 to 70.6% by weight in order to achieve both an effect of collecting ink (giving a high b value) and an ability to reduce the unliberated ink.

When the iodine value is high, the deinking agent would be adsorbed on the surface of ink almost uniformly to thereby form a thin adsorption layer (approximately 10 A). Then the effect of the surface potential (t-potential: -30 to -40 mV) of the ink per se becomes evident and thus the absolute surface charge density per unit area would not be lowered. As a result, the fine ink spot would hardly aggregate.

Therefore any deinked pulp of a high b value and contaminated with little unliberated ink cannot be obtained unless the numerical values fall within the ranges as specified in the present invention.

Next, the present invention wherein the components (a) and (b) are used together will be described in detail.

Namely, a particular embodiment of the present invention relates to a deinking method which comprises adding at least one surfactant (a) selected from the group consisting of compounds represented by the following general formulae (a-1) to (a-3) and a reaction product (a-4) obtained by adding an alkylene oxide to a mixture of a natural fat with a polyhydric alcohol exclusively in a stage of disintegrating (pulping) waste papers and adding a mixture (b) comprising higher fatty acid(s) having 8 to 24 carbon atoms or salt(s) thereof, wherein the average carbon atom number of the fatty acid(s) or salt(s) thereof ranges from 12.7 to 22.5, the content of higher fatty acid(s) having 20 to 24 carbon atoms or salt(s) thereof ranges from 9.6 to 70.6% by weight and the iodine value (IV) is not more than 45, exclusively in a mixing stage and/or flotation stage following the disintegrating stage:

$$R_1$$
-O-(-AO-)<sub>p</sub>-H (a-1)

wherein R<sub>1</sub> represents an alkyl or alkenyl group having 8 to 24 carbon atoms or an alkylphenyl group having an alkyl group carrying 6 to 14 carbon atoms; AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain when two or more kinds of the alkylene oxide are present; and p is such a number of 1 or above as to give the total molecular weight of from 800 to 10,000;

$$R_2$$
-COO-(-AO-)<sub>m</sub>-H (a-2)

wherein R<sub>2</sub> represents an alkyl or alkenyl group having 7 to 23 carbon atoms; AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain when two or more kinds of the alkylene oxide are present; and m is such a number of 1 or above as to give the total molecular weight of from 800 to 10,000; and

$$R_3$$
-O-(-AO-)<sub>n</sub>-SO<sub>3</sub>M (a-3)

wherein R<sub>3</sub> represents an alkyl, alkenyl or cycloalkyl group having 8 to 24 carbon atoms or an alkylphenyl group having an alkyl group carrying 8 to 12 carbon atoms;

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain when two or more kinds of the alkylene oxide are present;

n ranges from 1 to 5; and

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M represents an alkali metal or ammonium.

Now the present invention wherein the components (a) and (b) are used together, involving the aforesaid particular embodiment, will be described in detail. The component (b) is as described above.

Since the numerical values specified with respect to the compounds (a-1) to (a-4) relating to the present invention are critical ones, any compound similar thereto cannot exert any remarkable effect of the present invention unless it satisfies the specification of the present invention. Therefore the specification of the functional groups or numerical values of the compounds of the general formulae (a-1) to (a-3) in the present invention is very important.

The compound represented by the general formula (a-1) may be produced by a known method comprising adding an alkylene oxide to an alcohol. The total molecular weight of the alkylene oxide adduct of the alcohol ranges from 800 to 10,000, preferably from 1,000 to 4,000. This molecular weight is a number-average molecular weight determined by gel permeation chromatography (GPC) in terms of polyethylene glycol.

As the alkylene oxide to be added to an alcohol, ethylene oxide (EO), propylene oxide (PO), butylene oxide (BO) or a mixture thereof may be used. It is particularly preferable to use a mixture of EO with PO having a molar ratio of EO to PO of from 0.5/1 to 3/1. It is preferable, furthermore, that the alkylene oxide(s) are added via random addition so as to relieve foaming troubles in the papermaking and draining stages.

As the alcohol to be used for producing the compound represented by the general formula (a-1), those having an alkyl or alkenyl group carrying 8 to 24 carbon atoms or those having an alkylphenyl group having an alkyl group carrying 6 to 14 carbon atoms nay be used. Examples thereof include 1-octanol, 1-nonanol, 1-decanol, 1-undecanol, 1-dodecanol, 1-tridecanol, 1-tetradecanol, 1-pentadecanol, 1-hexadecanol, 1-hexadecanol, 1-hexadecanol, 1-hexadecanol, 1-tricosanol, 1-tetracosanol, 1-docosanol, 1-tricosanol, 1-tricosanol, 1-tricosanol, 1-docosanol, 1-tricosanol, 1-tricosanol, 1-docosanol, 1-tricosanol, 1-tricosanol, 1-docosanol, 1-tricosanol, 1-tricosanol, 1-docosanol, 1-tricosanol, 1-docosanol, 1-tricosanol, 1-docosanol, 1-tricosanol, 1-docosanol, 1-tricosanol, 1-docosanol, 1-docosanol, 1-tricosanol, 1-docosanol, 2-tridecanol, 2-tridecanol, 2-tridecanol, 2-tridecanol, 2-tridecanol, 2-tridecanol, 2-cotadecanol, 1-docosanol, 1-docosanol, 1-docosanol, 1-docosanol, 1-docosanol, 1-docosanol, 2-cotadecanol, 2-cotadec

As the compound represented by the general formula (a-1), those wherein  $R_1$  has 14 to 24 carbon atoms are preferable and those herein R is an alkyl group are further preferable.

The compound represented by the general formula (a-2) may be produced by a known method comprising adding an alkylene oxide to a fatty acid. Similar to the case of the compound represented by the general formula (a-1), the total molecular weight of the alkylene oxide adduct of the fatty acid ranges from 800 to 10,000, preferably from 1,000 to 4,000.

As the alkylene oxide to be added to the fatty acid, EO, PO, BO or a mixture thereof may be used. It is particularly preferable to use a mixture of EO with PO having a molar ratio of EO to PO of from 0.5/1 to 3/1. It is preferable, furthermore, that the allylene oxide(s) are added via random addition so as to relieve foaming troubles in the papermaking and draining stages.

As the fatty acid to be used for producing the compound represented by the general formula (a-1), those wherein R<sub>2</sub> has 7 to 23 carbon atoms may be used. Examples thereof include caprylic acid, pelargonic acid, capric acid, undecanoic acid, lauric acid, tridecanoic acid, myristic acid, pentadecanoic acid, palmitic acid, margaric acid, stearic acid, oleic acid, elaidic acid, linoleic acid, linolenic acid, stearolic acid, ricinolic acid, ricinelaidic acid, nonadecanoic acid, arachidic acid, heneicesanoic acid, behenic acid, brassidic acid, erucic acid, tricosanoic acid, tetracosanoic acid, coconut oil fatty acids, beef tallow fatty acids, palm oil fatty acids, tall oil fatty acids, rapeseed oil fatty acids and fish oil fatty acids.

As the compound represented by the general formula (a-2), those wherein  $R_2$  has 11 to 23 carbon atoms are preferable and those wherein R is an alkyl group is still preferable.

The compound represented by the general formula (a-3) may be produced by a known method comprising adding an alkylene oxide to an alcohol having an alkyl or alkenyl group carrying 8 to 24 carbon atoms or an alkylenenyl group having an alkyl group carrying 8 to 12 carbon atoms followed by the sulfonation and neutralization of the obtained adduct. Either a straight-chain alkyl group or a cyclic one may be used. Examples of such alcohols include 1-octanol, 1-nonanol, 1-decanol, 1-undecanol, 1-dodecanol, 1-tridecanol, 1-pentadecanol, 1-hexadecanol, 1-heptadecanol, 1-octadecanol, 1-nonadecanol, 1-eicosarol, 1-heneicosanol, 1-docosanol, 1-tricosanol, 1-tetracosanol, 2-nonanol, 2-decanol, 2-undecanol, 2-tridecanol, 2-tetradecanol, 2-pentadecanol, 2-hexadecanol, 2-heptadecanol, 2-octanol, 2-octanol,

1-ol, 2-dodecen-1-ol, 2-undecen-1-ol, 2-tetradecen-1-ol, 2-pentadecen-1-ol, 2-hexadecen-1-ol, 2-octadecen-1-ol, 8-nonen-1-ol, 10-undecen-1-ol, 11-dodeten-1-ol, 12-tridecen-1-ol, 15-hexadecen-1-ol, oleyl alcohol, elaidyl alcohol, linoleyl alcohol, linolenyl alcohol, eleostearyl alcohol, ricinoyl alcohol, cyclononanol, cyclodecanol, cycloundecanol, cyclodecanol, cyclotridecanol, cyclotridecanol, cyclotridecanol, cyclotridecanol, cyclocesanol, cyclopentadecanol, cyclohexadecanol, cyclohexadecanol, cyclocesanol, cyclocesanol, octylphenol and nonylphenol. An alcohol having an alkyl group is preferable therefor.

As the alkylene oxide to be added to the alcohol, EO, PO, BO or a mixture thereof may be used. It is particularly preferable to use a mixture of EO with PO having a molar ratio of EO to PO of from 1/1 to 5/1. The addition mole number of the alkylene oxide may preferably range from 1 to 5.

In the general formula (a-3), M represents an alkali metal, such as sodium or potassium, or ammonium.

The component (a-4) relating to the present invention is an alkylene oxide adduct of a mixture of a natural fat with a polyhydric alcohol. In the present invention, the mixing ratio of the natural fat to the polyhydric alcohol in the component (a-4) and the addition mole number of the alkylene oxide are highly important. It is desirable to add 5 mol or more, still preferably from 20 to 100 mol, of the alkylene oxide per mol of the natural fat.

The mixing ratio, on a molar basis, of the natural fat to the polyhydric alcohol may preferably range from 1/0.1 to 1/3, still preferably from 1/0.2 to 1/2.

As the alkylene oxide to be added to the mixture of the natural fat with the polyhydric alcohol, EO, PO, BO or a mixture thereof may be used. It is particularly preferable to use EO and PO. Although different alkylene oxides may be either mixed with each other before the addition (random addition) or successively added (block addition), the random addition is preferred by taking foaming troubles in the papermaking and draining stages into consideration.

Examples of the natural fat to be used in the component (a-4) of the present invention include vegetable oils such as coconut oil, palm oil, olive oil, soybean oil, rapeseed oil and linseed oil, animal fats such as lard, beef tallow and bone oil, fish oils, those obtained by hardening or semi-hardening the above fats and those obtained during the refining process of these fats.

Examples of the polyhydric alcohol to be used in the component (a-4) of the present invention include ethylene glycol, propylene glycol, trimethylene glycol, butylene glycol, 1,6-hexane glycol, 2-ethylbutane-1,2,3-triol, glycerol, trimethylolpropane, trimethylolethane, 1,2,4-butanetriol, 1,2,6-hexanetriol, 1,1,1-trimethylolhexane, tetramethylolcyclohexanol, diglycerol, mannitol, pentaerythritol, erythritol, arabitol, sorbitol, D-glycero-D-galactoheptose, D-glycero-D-glucoheptose, D-glycero-D-mannoheptose, D-glycero-D-mannoheptose, D-glycero-D-galactoheptitol, D-erythro-D-galactitol, D-glycero-D-mannoctulose, D-erythro-L-gulononulose, cellobiose, maltose, lactose, gentianose, cellottiose and stachyose. It is particularly preferable to use di- to hexahydric alcohols.

The components (a-1) to (a-4) relating to the present invention may be added exclusively in the disintegrating (pulping) stage whereas the component (b) may be added exclusively in the mixing and/or flotation stages following the disintegrating stage to thereby achieve excellent effects. Although a deinked pulp of a certain degree of qualities may be obtained by adding a mixture of the components (a) and (b) or by adding the components (a) and (b) in the reverse order, it is disclosed herein that the method of the present invention makes it possible to obtain a deinked pulp which has a high whiteness and a high b value and is contaminated with little unliberated ink.

In the present invention, the preferable ratios of each of the components (a-1) to (a-4) to the component (b) to be added exist. More specifically the ratios, on a weight basis, of these components may preferably fall within the ranges as specified below:

(a-1)/(b) = 10/90 - 90/10, preferably 40/60 - 60/40;

(a-2)/(b) = 10/90 - 90/10, preferably 40/60 - 60/40;

(a-3)/(b) = 5/95 - 30/70, preferably 10/90 - 25/75; and

(a-4)/(b) = 10/90 - 70/30, preferably 20/80 - 50/50.

When each weight ratio is above the lower limit as specified above, the liberation of ink from cellulose is further improved and thus the amount of the unliberated ink is decreased. When this weight ratio is below the upper limit as specified above, on the other hand, the ability to collect ink is enhanced and thus the obtained deinked pulp has a higher whiteness and a higher b value. When the weight ratio of (a-3)/(b) is below the upper limit, in particular, the foam breakage in the flotation reject is highly improved and thus a back flow of the flotation reject toward the flotation cell might hardly occur. As a result, the deinking ratio is elevated and a deinked pulp of a high whiteness and a high b value can be obtained.

The total amount of the components (a) and (b) to be added in the present invention may preferably fall within such a range that the workability of the process is not deteriorated and the procedure can be economically performed. It may preferably range from 0.03 to 1.0% by weight based on the waste papers.

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The invention will be below illustrated in reference to its examples of (b) and its mixture with (a-1), (a-2), (a-3) or (a-4).

Examples 1 to 4 of (b) provide invention products 1 to 53 in Table 1-1 to 8-1. Example 1

In this Example, a deinking agent was added as a whole in the pulping stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.8% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.5% (based on the starting material) of each of the deinking agents listed in Table 1 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the pulp concentration reached 23% and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

The term "b value" as used herein means the value of b of the Lab space indication system in accordance with Hunter's color difference formula. The relation thereof with the tristimulus values (X, Y and Z) is as follows:

# $b = 7.0 (Y - 0.847Z)/\sqrt{Y}$

As the above equation shows, the b value is a function of Y and Z. A positive b value means the intenseness of yellowness, while a negative one means that of blueness.

Table 2 shows the deinking performances of the deinking agents of the present invention achieved by varying the average carbon atom number and the content of the higher fatty acids having 20 to 24 carbon atoms.

Each of the deinking agents listed in Table 1, is prepared by blending individual fatty acids with each other in such a manner as to give a composition of a specified carbon atom number. The deinking agent No. 19 comprises stearic acid.

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Table 1-f(No. 1)

		-			_	_	_	<u> </u>	_	_	-		
Content of	fatty acids	9.6	1.0	12.5	12.0	16.5	13.0	30.5	37.3	54.0	62.0	1.19	70.0
	3	:	0.1	0.1	0.1	9.1	9.5	=	1.1	10.4	23.5	19.1	69.4
	3	0.05	0.03	0.05	0.05	0.0	0.05	0.05	0.05	0.1	0.1	0.1	0.1
	ڻ	9.9	6.9	7.5	9.6	10.7	13.4	13.4	18.1	26.9	21.2	20.6	0.2
	t, 5	0.03	0.08	0.05	0.05	0.05	0.08	0.08	0.08	0.1	0.1	0.1	0.1
	<b>*</b>	4.5	4.7	1.8	5.1	5.6	8.8	5.3	15.4	16.5	17.9	4.2	0.2
=	°,	0.05	0.03	0.02	0.03	0.0	90.0	0.03	0.05	0.1	0	0	11.6
ion (vt	C,0	3.6	0.4	0.5	9.6	34.1	\$2.0	74.8	47.0	9.2	17.6	32.9	10.4
mposit	כיי	0.04	0.01	0.01	10.0	0.06	0.04	0.01	0.03	٥	0	•	0
fatty acid carbon No. composition (wt.1)	C,1	0.2	0.3	22.9	11.0	36.7	10.4	0.1	14.6	36.1	19.4	2.9	0
carbon	C,1	9.00	٥	0.03	0.03	0	0	0	•	٥	۰	0	•
y acld	с''	0.3	30.7	11.5	29.62	0.3	12.4	0.1	0.3	0.1	9:1	0.1	0
rati	C <sub>0.1</sub>	0.05	0.0	0.03	0.03	0	0	•	٥	۰	٥	0	0
	C1,	63.6	40.5	10.5	3.7	2.1	2.1	1.0	0.3	0.2	0.1	. 0	0
	C <sub>II</sub>	90.0	0.01	0.01	0.01	0	0	0	0	٥	0	0	0
	C.	21.1	0.1	2.3	0.8	7.0	3.2	0	0.2	0.1	0	0	•
	ů	0.04	0.05	0.05	0.01	٥	0	0	0	c	٥	0	0
	ئ	?	16.1		3.6	7.5	7:1	1.0	0.2	:	•	0	0
lodine value	Š	0.1	2.0	6.0	<u>;</u>	2.1	3.5	1.9	5.4	4.3	1.0	7.6	6.2
Average C atcm Ho.		2:2	13.1	14.5	15.6	16.9	17.4	10.2	18.9	19.3	20.4	31.2	6.65
	<u>(</u> 9	-	~	^	-	^	۰	٦	0	•	9	=	7,
			product s										

		┡-										1					ľ		ľ	
	*10m 110.	**			Ī	Ì	Ì	ž	ratty acid carbon	og 150		20 15	No. composition (wt.1)	=						Content of
		2.0	ű	ບໍ	<b>.</b> "	ניי	Ĵ	j.	J.	Ĵ	J.	j.	C,	6,1	ر»	<del>ن</del>	ر."	ر.,	ڻ	falty acids
=	3.3	1.0	9.1	0	0	٥	0	٥	۰	0	۰	۰	•	-	-	•	-	۰	<u>•</u>	10.9
product s	19.4	2.7	7.7	0.04	13.4	0.06	6.9	0.04	3.2	0.03	1.6	0.03	0.1	0.02	2	•	=	6	=	2.5
2	2.5	1:3	?	0.08	33.5	0.03	\$1.8	0.03	6.3	0.04	0.3	0.01	3.4	0.03	4.3	0.0	1.9	0.03	5	10.6
=	12.0	. 3.4	=	0.0	1:6	0.06	16.8	0.04	2.6	0.02	2.1	0.03	1.4	10.0	0	•	0	٥	2.	-;
=	13.2	6.3	16.0	0.0	9.1	0.01	77.7	0.04	45.9	10.0	0.9	0.02	0.8	0	3.8	0.03	8.8	0.03	0.1	9.5
=	17.3	£.2	1.2	•	3.3	0	1.1	0	12.6	0	11.2	0.03	57.7	0.08	0.1	0	2.3	٥	• .	•:
2	10.0	1.3	٥	٥	۰	٥	•	۰	0	•	0	0	100	•	•	0	•	۰	-	•
2	. 19.4	2.0	۰	۰	3	٥	0.0	۰	13.7	٥	12.0	0	0.2	0.1	27.4	0.03	35.4	0.03	5.8	70.7
~	23.4	5.1	۰	۰	•	•	•	0	۰	0	•	0	0	0	0	0	1.98	•	13.3	100
≈	23.9	7.3	۰	٥	٥	۰	٥	۰	0	۰	۰	٥	1:1	٥	0.3	0.08	0.4	0.03	1.06	91.9
23	24.0	1.0	•	•	•		•	0	•	•	•	•	•	۰	۰	•	•	•	901	100

Table 1-((No. 2)

Table 2-1

	Deinking age	nt No.	Qualities	of deinked pulp
5			b value (%)	unliberated ink No.
	Invention	1	8.66	8
•	products	2	. 8.79	10
10		3	8.61	9
. •	*	4	9.06	8
		5	8.97	9
		6	8.91	10
15		7	9.20	7
	+	8	9.45	5
		9	9.22	. 7
20		10	9.02	10
		11	9.03	8
•		12	8.74	. 9
25	Comparative	13	6.56	29
	products	14	6.53	34
		15	6.61	33
		16	6.08	33
30 ·		17	6.54	31
		18	6.24	34
		19	6.82	30
9 <b>5</b>	;	20	7.11	42
		21	7.63	. 31
		22	7.86	42
10		23 .	7.85	48
· <del>·</del>				

# Example 2

In this Example, a deinking agent was added in portions in the pulping and chemical mixing stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.1% (based

disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 22%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Table 3 were added thereto. After adjusting the pulp concentration to 23% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at

30°C for 10 minutes. During the flotation process, 0.4% (based on the starting material) of CaCl2 was

on the starting material) of each of the deinking agents listed in Table 3 were added thereto. After

added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (ca/Mg = 8/2 by mol).

The b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100  $\times$  magnification).

Table 4 shows the deinking performances achieved by changing the iodine values (IV) of the deinking agents.

Each of the deinking agents listed in Table 3 is prepared by blending individual fatty acids with each other in such a manner as to give a composition of a specified carbon atom number.

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	i						I	ı													
		Average C	lodine value						Pate	y seld	carbon	fetty acid carbon No. composition (wt.1)	mostel	on let							Content of
	<u></u>		(IV)	J	ر,	C.e	<b>C</b> 11	C <sub>11</sub>	C <sub>1</sub> 3	, t	ď	ا. ت	r'b	15	J.	3	ű	ű	3	ئ	fatty acids
	~	12.0	1.7	0.2	0.04	11.12	90.0	63.6	0.03	0.2	90.0	0.2	0.04	3.5	0.03	÷:	9.03	6.2	0.05	=	10.9
product s	٦	11.5	1.3	•	0.03	2.3	0.01	10.5	0.02	11.5	0.03	22.9	0.01	0.5	0.02	1.0	0.05	7.5	0.05	0.1	12.5
	×	13.6	10.9	3.6	10.0	0.8	0.01	3.7	0.03	29.6	0.02	14.8	0.01	9.0	0.03	5.1	0.03	9.6	0.03		2.≅
	~	16.0	. 26.6	3.2	۰	1.0	۰	2.1	•	0.3	0	36.7	90.0	34.1	0.04	3.6	0.08	10.7	0.08	0.1	16.5
	28	18.9	4.0	2.5	٥	9.2	٥	0.3	0	0.3	0	14.6	0.03	47.0	0.08	15.4	0.05	10.1	0.03	3.7	19.3
	23	20.4	31.5	•	•	0	0	0.1	0	0.1	0	19.4	0	17.6	0	17.9	0.1	21.12	0.1	23.5	62.0
	۲	6.55	\$.2	•	٥	0	•	٥	•	0	٥	0	0	10.4	11.6	0.2	0.1	0.2	0.1	69.4	10.0
	31	. 12.0	6.34	0.5	0.04	1.18	90.0	63.6	0.08	0.2	90.0	0.2	0.04	3.8	0.03	. 1.5	0.08	6.2	0.05	0.1	10.9
product a	î	11.5	63.0	:	0.03	2.3	0.01	10.5	0.02	41.5	0.02	22.9	10.0	0.9	0.03	•:•	0.05	1.5	0.05	0.1	12.5
	33	13.6	19.1	3.6	0.01	9.5	0.01	3.7	0.02	23.62	9.03	4.0	10.0	9.0	0.03	5.1	0.03	9.6	0.03	0.1	12.0
		16.0	95.4	3.2	0	1.0	0	1.2.	•	0.0	۰	36.7	0.06	34.1	0.04	3.6	0.08	10.1	0.08	0.1	16.8
	35	10.9	130.1	0.5	0	9.2	0	0.3	٥	6.9	۰	=	0.03	47.0	0.03	15.4	0.05	10.1	0.05	3.7	39.3
	36	20.4	171.3	۰	•	•		0.1	•	0.1	•	19.4	۰	17.6	•	17.9	0.1	21.2	7.	23.5	62.0

Table 4-1

Deinking ager	nt No.	Qualities	of deinked pulp
4	٠	b value (%)	unliberated ink No.
Invention	24	8.90	. 7
products	25	8.94	8
•	26	8.83	9
	27	8.79	9
	28	9.46	5
•	29	9.01	7
•	30	9.04	7
Comparative	31	7.16	29
products	32	7.23	32
	33	7.05	. 35
	34	6.85	36
•	35	6.25	51
	36	6.47	. 48

#### Example 3

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In this Example, a deinking agent was added in portions in the pulping and kneading stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.3% (based on the starting material) of each of the deinking agents lasted in Table 5 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached. 22%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Table 5 were added thereto. After adjusting the pulp concentration to 23% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 10° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 6 shows the deinking performances of the deinking agents.

The deinking agents No. 37 to No. 40 listed in Table 5 were respectively obtained from hardened fatty acids of cod, sardine, saury and mackerel fish oils, while the deinking agent No. 41 was obtained by mixing the deinking agent No. 37 with the deinking agent No. 38 at a weight ratio of 50/50. The deinking agents No. 42 to No. 44 were obtained by changing the iodine value (IV) of the deinking agent No. 37. Further, the deinking agents No. 45 to No. 49 respectively comprised stearic acid, myristic acid, commercially available stearic acid [Lunac S-40; a product of Kao], hardened beef tallow fatty acids and sardine oil fatty acids (IV = 175).

Table 5-1

	I																				
		Average C	value	_ ]					Fatt	y seld	carbon	Fatty acid carbon Ho. composition (wt.1)	positi	lon (vt	١٠.						Content of
	É		(iv	Ĵ	ů	<b></b>	ر"	Ĵ,	C,1	ړ.	ů,	j	ر"	<b>"</b> "	<b>3</b> 5	ž,	ڻ	J	ٿ	ڻ	(atty acids
	=	10.5	0.3	٥	0	0	0	0	•	6.1	•	36.9	•	3.8	•	5.5	•	2.5	•	-	= -
product s	Ä	10.5	0.3	0	0	0	0	۰	۰	6.3	۰	25.0	۰	36	۰	20.2	•	=	-	=	40.9
	ŝ	19.0	. 0.3	٥	0	0	0	0	•	6.9	۰	19.0	۰	23.0	•	2.2	•	27.5	•	0.0	51.1
	\$	19.0	0.3	•	•	0	0	0	•	6.0	0	22.7	٥	23.0	۰	8.02	•	26.7	۰	•	47.5
	₹	10.5	0.3	٥	٥	0	0	0	0	4.2	0	31.0	0	23.6	0	19.0	۰	<u>≈</u>	•	6.	£.5
	÷	19.5	23.5	0	0	0	0	0	•	1.9	.0	36.9	0	19.8	0	17.0	۰	\$.5	۰	•	÷.:
	3	. 10.5	44.0	۰	0	0	0	0	•	1.9	0	36.9	0	19.5	0	17.0	٥	23.9	0	•	4.1
	٤	10.5	47.5	٥	۰	0	0	0	•	1.9	0	36.9	٥	19.8	٥	17.0	0	23.9	0	0	41.7
product s	Ş	18.0	0.3	0	۰	0	0	0	•	0	0	0	٥	100	0	0	0	•	0	•	0
	\$	14.0	0.3	•	0	0	۰	0	•	100	۰	•	0	0	0	0	0	0	0	0	0
	•	17.0	0.3	0	•	•	0	0	•	0	•	0.0	0	92.0	0	0	0	0	.6	0	0
	=	16.8	0.3	0	0	0	0	0.2	0.1	29.3	1.0	6.2	0.1	63.0	0.1	0.1	٥	.0	0	0	0.1
	97		136.0	٠	٠	•	7	ļ	•	,	•		•	3, 4,	•	, 04	-	:	•	7 1	• 07

Table 6-1

Deinking age	nt No.	Qualities	of deinked pulp
	•	b value (%)	unliberated ink No.
Invention	37	9.52	. 2
products	38	9.65	2
	39	9.64	2
	40	9.63	2
	41	9.62	3
•	42	9.46	. 4
<u>.                                      </u>	43	9.33	4
Comparative	44	7.82	. 12
products	45	7.52	. 29
	46	7.01	. 32
	47	7.43	28
•	48	7.32	.31
	49	6.02	54

#### Example 4

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In this Example, a deinking agent was added in portions in the pulping stage and before the flotation stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.2% (based on the starting material) of each of the deinking agents listed in Table 7 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 22%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3 and 3.5% (based on the starting material) of 30% hydrogen peroxide were added thereto. After adjusting the pulp concentration to 23% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator fox 30 seconds. Then 0.3% (based on the starting material) of each of the deinking agents listed in Table 7 was added to the obtained pulp slurry. After diluting with water so as to give a pulp concentration of 1%, the slurry was subjected to flotation at 30°C for 10 minutes. After the completion of the flotation, the pulp slurry was concentrated with a 60-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 40° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyser (100 x magnification).

Table 8 shows the deinking performances of the deinking agents.

In Table 7, all of the fatty acids contained in the deinking agents were in the form of sodium salts. Each of the deinking agents No. 50 to No. 53, is prepared by blending individual sodium salts of the fatty acids with each other in such a manner as to give a composition of a specified carbon atom number. The deinking agent No. 54 comprised sodium stearate.

The deinking agents Nos. 60 and 61 are each prepared by blending calcium salts of fatty acids with each other so as to produce a composition having the specified carbon atoms number. The deinking agent

No. 54 comprised sodium stearate and No. 62 comprised calcium stearate.

The hardness of the employed water was adjusted to 40° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 8 shows the deinking performances of the deinking agents.

In Table 7, all of the fatty acids contained in the deinking agents were in the form of sodium salts. Each of the deinking agents No. 50 to No. 53, is prepared by blending individual sodium salts of the fatty acids with each other in such a manner as to give a composition of a specified carbon atom number. The deinking agent No. 54 comprised sodium stearate.

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Table 7-1

		Average C.	Jodine velue						7.5	ty actd	carbon	fatty acid carbon Ho. composition (wt.1)	positi	ion (vt.	3						Content of
//	6		(11)	°,	ڻ	<b>"</b> "	ניו	<b>3</b>	6,1	·*	C,	ď	ر"،	C,	C,	<b>6</b> 2	ű	"2	<b>c</b> ,,	ť	fatty acids (vt.1)
	ž	15.0	0.4	0	3	٥	٥	۰	۰	7.8	۰	14.0	۰	17.4	۰	10.1	۰	20.3	0	22.4	60.0
product s	2	16.0	5.3	0	٥	ţ	0	10.2	0	5.2	0	0	0	60.3	0	0.0	٥	16.3	٥	-	24.3
	22	17.7	0.9	4.2	0	10.3	0	6.8	0	1.9	•	9.0	0	10.2	0	19.0	0	18.3	٥	13.1	\$1.2
	53	18.9	3.2	0	0	0	0	0	0	0	•	16.9	٥	42.3	0	19.0	0	20.9	٥	0.1	40.0
	15	18.0	0	•	0	0	0	•	0	0	•	۰	0	100	٥	0	0	0	٥	۰	0
Comparative	35	Steary	alcohol (EO), (PO), random adduct	(EO) 11 (P	0)	ndom ad	duct.														
	36	stearic	acid (EO), (PO), block adduct	, (04),	block	* adduct															
	3,1	nony lphe	nol (E0), adduct*	adduct	•																
	\$	p unipos	odecylbentenesulfonate	1 C D D D D	Ifonat																
	\$\$	unipos	auryl sulfate	fate																	
Invention	09	19.8	0.4	0	0	0	0	•	0	7.8		14.0	0	17.4	0	16.1	0	20.3		22.4	
	<b>=</b>	18.0	S.2				0	10.2		5.2	0	-	0	60.3	0	8.0	0	16.3	c	<u></u>	24.3
Comparative 62	3	18.0	0.0	0	c	•	c	c	0	•	0	0	•	901	0	.0	c	0	<b>c</b>	:_ !	0

ling. \* ED: ethylane oxide; PD: propylane oxide; the number represents the addition mole number.

Hos. 51 and 52: calcium salts.

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Table 8-1

Deinking agent No	<b>)</b> .	Qualities	of deinked pulp.
		b value (%)	unliberated ink No.
Invention process	50	9.42	5
	51	9.43	5
	52	9.72	2
	53	9.61	2
Comparative	54	7.51	28
products	55	7.32	25
•	56	7.01	23
	57	6.54	41
	58	6.50	35
•	59	6.42	43
invention	60	9.40	5
	61	9.41	. 5
Comparative product	62	7.48	30

As the above Examples show, a deinked pulp having a high b value and contaminated with little unliberated ink can be obtained by using a mixture containing higher fatty acid(s) having 8 to 24 carbon atoms or salt(s) thereof, wherein the average carbon atom number of the fatty acids in the mixture ranges from 12.7 to 22.5, the content of higher fatty acid(s) having 20 to 24 carbon atoms or salt(s) thereof ranges from 9.6 to 70.6% by weight and the iodine value (IV) is not more than 45, as a deinking agent.

#### Example 5

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Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 1.5% (based on the starting material) of caustic soda, 1.3% (based on the starting material) of sodium silicate No. 3, 1.5% (based on the starting material) of 30% hydrogen peroxide and the deinking agents listed in Table 9 were added thereto. After disintegrating at a pulp concentration of 15% at 60°C for 15 minutes, the mixture was aged at 55°C for 120 minutes. After diluting with hot water so as to give a pulp concentration of 5%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 50°C for 7 minutes. After the completion of the flotation, the pulp slurry was concentrated with a 60-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The b value and brightness of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification). Table 10 shows the deinking performances.

Table 9

No.	Deinking Agent	Amount (wt%)
1	higher fatty acid of the present invention	0.6
2	higher fatty acid sodium dodecylbenzenesulfonate	0.5 0.1

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Table 10

No.	Brightness (%)	b Value (%)
1	56.5	10.1
2	54.6	8.2

#### 10 Example 6

In this Example, a deinking agent was added as a whole in the pulping stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.8% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.5% (based on the starting material) of each of the deinking accents listed in Tables 1 to 3 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the pulp concentration reached 23% and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and than subjected to flotation at 30°C for 10 minutes. During the rotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 11 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 12 shows the component (a) and Table 13 shows the weight ratio of the components (b) to (a). Further, Table 14 shows the deinking performances.

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Table 11

Content of Ca Cat (atty acids (atty acids ... 3 9.1 0.03 0.03 9.0 10.1 20.6 9.8 0.05 0.05 0.03 0.02 0.05 تّ 2. ئ 0.02 0.0 ٿ composition (wt.1) 17.0 ٿ ÷ : 36.7 14.6 0.2 ٿي €. Fatty sold carbon 90.0 0.03 ٿ 0.2 29.6 70.0 6.3 : J 0.05 0.01 0.03 5 63.6 **6**.5 3 9.00 0.0 0.0 Ĵ 0.04 ů 0.2 J rodine value Average C atom No. 12.8 13.1 16.0 15.6 16.9 20.4

Table 12

Deinking at No.	gent	Component (a)
	1	C <sub>12</sub> H <sub>25</sub> O(EO) <sub>10</sub> (PO) <sub>5</sub> H
Invention products	2	C <sub>18</sub> H <sub>27</sub> O(EO) <sub>100</sub> H
	.3	C,H,, -(O- O (EO) 6 (PO) 4H
· ·	4	C <sub>16</sub> H <sub>23</sub> O (PO) 10 (EO) 10H
	5	C <sub>18</sub> H <sub>35</sub> O (EO) <sub>50</sub> (EO) <sub>30</sub> H
	6	C <sub>8</sub> H <sub>1</sub> , -(O)O (EO) <sub>5</sub> (PO) <sub>10</sub> H
	7	C;4H2,0 (EO) ;0 (EO) 15H
	8	C;cH2:O(EO);0 (BO);H

	Table	13
Deinking ag	ent No.	Weight ratio (b)/(a)
Invention	1	90/10
products	· 2	80/20
	3	70/30
	4	65/35
	5	62/38
·	6	53/47
•	7	45/55
*	8	40/60

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Table 14

Deinking ag	ent No.	Qualit	ies of deinked p	ulp
		whiteness (%)	b value (%)	unliberated ink no.
Invention	1	55.2	9.97	5
products	2	55.3	9.98	7
·	3	55.4	9.86	7
	4	55.7	10.1	6
	5	56.3	10.2	4
	6	55.6	10.1	6
	7	55.5	10.0	. 5
	8	55.4	10.0	5

Example 7

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In this Example, a deinking agent was added in portions in the pulping and chemical mixing stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.1% (based on the starting material) of each of the deinking agents listed in Tables 15 to 17 were added thereto. After disintegrating at a pulp concentration of 15% at 45 °C for 12 minutes the mixture was aged at 55 °C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reacted 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Tables 15 to 17 were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30 °C for 10 minutes. During the flotation process, 0.4% (based on the starting material) of CaCl2 was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 15 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 16 shows the component (a) and Table 17 shows the weight ratio of the components (b) to (a). Further, Table 18 shows the deinking performances.

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		;								Tabl	Table 15										•
		Average C	fodine value				-	- 2	1	ty acto	Carbon	Fatty acid carbon No. composition (wt.1)	Postt	2 8	=						
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Thurst Los	=	22.8	40.1	0.2	0.04	23.1	90.0	63.6	63.6 0.05	0.2	0.2 0.06		0.0		:	:	<del>!</del>	Ī	Ī	İ	
or conference	=	7 71		:	3	<u>.                                    </u>	_								3	4.3 0.05	4	7.9	0.03	0.1	10.9
	•			•	9.0	9.5	0.01	1.1	3.7 0.02	29.6 0.02		::	0.0	0.6 0.03	0.03	-	2	•	,,,		
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	٦	22.3	6.7	•	٥	٥	•	•	0	٥		•	٠			;		_	+		
						ĺ	Ī	I		Ī	İ	1	ŀ	i	1	7:0	_1	?		69.4	0.00

Table 16

Deinking ac	gent	Component (a)
	17	C12H25O (EO) 50H
Invention products	18	C18H37O (EO) 22 (PO) 11H
	19	C12H32O(PO)10(EO)10H
	20	C <sub>16</sub> H <sub>33</sub> O (PO) <sub>12</sub> (BO) <sub>6</sub> H
	21	C <sub>5</sub> H <sub>25</sub> -(PO) (EO) (PO) (H

Table 17

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Table 18

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Deinking ag	ent No.	Qualit	ies of deinked p	ulp .
		whiteness (%)	b value (%)	unliberated ink no.
Invention	17	55.8	10.3	4
products	18	55.9	10.1	5
	19	56.5	10.3	4
_	20	55.7	10.4	.6
	21	55.8	10.2	6

# Example 8

In this Example, a deinking agent was added in portions in the pulping and kneading stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.3% (based on the starting material) of each of the deinking agents listed in Tables 19 to 21 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Tables 19 to 21 were added thereto. After

adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30° C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to gave a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 10° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 19 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 20 shows the component (a) and Table 21 shows the weight ratio of the components (b) to (a). Further, Table 22 shows the deinking performances.

The components (b) listed in Table 19 were cod, sardine, saury and mackerel oil fatty acids (No. 26 - No. 29) and the one of No. 30 was a mixture of No. 26 and No. 27 at a weight ratio of 50/50. Those of No. 31 to No. 33 were prepared by varying the iodine value (IV) of the one of No. 26.

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				17.	20.2		22.7	20.6	•		
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	fatty acid carbon No. composition (wt.1)	J		26.5	25.0		19.0	1.52	9.15	2	
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		//		Invention	products						

Table 20

Deinking agent Component (a) No. 26 C12H250 (EO) 10 (PO) 5H Invention C18H370 (EO) 70 (PO) 30H 27 products C, -(D- 0(EO), (PO), H 28 29 C16H33O (PO) 15 (EO) 15H 30 C<sub>18</sub>H<sub>35</sub>O (EO) <sub>22</sub> (BO) <sub>10</sub>H 31 C<sub>14</sub>H<sub>29</sub>O (EO) <sub>30</sub> (PO) <sub>15</sub>H C<sub>14</sub>H<sub>29</sub>O (EO) 30 (PO) 15H 32

Table 21

Deinking agent No. Weight ratio (b)/(a) Invention 26 80/20 products 27 70/30 28 65/35 29 60/40 30 50/50 31 85/15 32 85/15

Table 22

Deinking ag	gent No.	Qualit	ies of deinked p	ulp
ž		whiteness (%)	b value (%)	unliberated ink no.
Invention	26	57.5	10.6	3
products	27	57.6	10.7	4
	28	57.8	10.8	2 .
*	29	57.5	10.7	3
	30	57.4	10.8	3
	31	57.5	10.8	2
	32	57.5	10.3	5

Example 9

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In this Example, a deinking agent was added in portions in the pulping stage and before the flotation stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.2% (based on the starting material) of each of the deinking agents listed in Tables 23 to 25 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3 and 3.5% (based on the starting material) of 30% hydrogen peroxide were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. Then 0.3% (based on the starting material) of each of the deinking agents listed in Tables 13 to 15 was added to the obtained pulp slurry. After diluting with water so as to give a pulp concentration of 1%, the slurry was subjected to flotation at 30 °C for 10 minutes. After the completion of the flotation, the pulp slurry was concentrated with a 60-mesh wire to thereby give a concentration of 4%. Then it was diluted with water So as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 40° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 23 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 24 shows the component (a) and Table 25 shows the weight ratio of the components (b) to (a). Further, Table 26 shows the deinking performances.

In Table 23, all of the fatty acids as the component (b) contained in the deinking agents were in the form of sodium salts. Each of the components (b) No. 39 to No. 42 is prepared by blending individual sodium salts of the fatty acids with each other in such a manner as to give a composition of a specified carbon atom number.

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Invention products	ę	10.0	3.3	٥	0	c	•	0 16.2 0 5.2 0	•	5.2	0	0	۰	0 60.3 0 8.0 0 16.3	•	0.0	0	16.3	۰	c	24.3	
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	~	18.9	::	0	0	0	•	•	0	•	0	0 16.9 0 42.3 0 19.8 0 20.9 0	•	42.3	•	19.8	۰	20.9	٦	ءَ	40.4	

Table 24

Deinking ag	ent	Component (a)
	39	C <sub>12</sub> H <sub>25</sub> O (EO) <sub>10</sub> (PO) <sub>5</sub> H
Invention products	40	C <sub>16</sub> H <sub>37</sub> O (EO) <sub>20</sub> (PO) <sub>16</sub> H
	41	C <sub>9</sub> H <sub>29</sub> ————————————————————————————————————
	42	C <sub>10</sub> H <sub>21</sub> O (EO) <sub>10</sub> (BO) <sub>5</sub> H

Table 25

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Deinking agent No. Weight ratio (b)/(a)

Invention 39 90/10

products 40 80/20

41 70/30

42 60/40

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Table 26

Deinking age	ent No.	Qualit	ies of deinked p	ulp
		whiteness (%)	b value (%)	unliberated ink no.
Invention	39	57.8	10.7	2
products	40	57.6	10.8	4
	41	57.6	10.8	2
	42	57.7	10.8	3

## Example 10

In this Example, a deinking agent was added as a whole in the pulping stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a beach disintegrator. Then water, 0.8% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.5% (based on the starting material) of each of the deinking agents listed in Tables 1 to 3 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the pulp concentration reached 23% and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so

as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use or CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 27 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 28 shows the component (a) and Table 29 shows the weight ratio of the components (b) to (a). Further, Table 30 shows the deinking performances.

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Table 27

		Average C	lodine value						rett	y seld	Fatty sold carbon	. co	Mo. composition	on (wt.1)	=						Content of
//	Ē		(14)	<b>3</b>	C,	ر و	ניו	υ,	<b>1</b> '5	۲,	C,1	C <sub>1</sub> ,	C,,	°13	ڻ	ď	5	ڻ	ئ	ئ	(atty acids
	-	12.0	7:1	6.2	0.04	21.1	90.0	9.6	0.05	0.2	90.0	0.2	0.04	3.0	0.08	\$	0.03	5.9	0.03	0.1	9.6
product s	~	13.1	1:1	1.8.1	6.6	0.1	10.0	49.5	0.01	30.7	•	0.3	0.01	0.4	0.03	4.7	0.03	6.9	0.03	0.1	11.0
	_	15.6	2.9	3.6	0.01	0.5	0.01	1.1	0.02	29.6	0.02	14.6	10.0	9.0	0.03	1.6	0.03	9.6	0.03	0.1	12.0
	-	16.8	3.0	3.2	۰	1.0	•	1.1	0	0.3	•	36.7	90.0	34.1	0.04	9.6	0.03	10.9	0.03	0.1	16.5
	^	17.4	6.8	1.2	٥	3.2	0	1.1	0	12.4	•	10.4	0.04	\$2.0	90.0	9.6	0.0	11.4	0.03	0.5	17.0
	_	19.9	7.6	7:0	٠	0.2	0	0.3	•	0.3	•	14.6	0.03	47.0	0.03	13.4	0.03	18.1	0.03	3.1	37.3
	•	20.4	3.4	•	•	c	0	0.1	0	0.1	•	19.4	•	17.6	۰	17.9	0.1	21.2	9.1	23.5	62.0
	•	71.2	8.9	0	٥	0	0	0	۰	0.1	٥	2.5	۰	33.9	•	4.2	7.	20.6	9.1	39.1	64.1
	•	22.3	1.3	•	۰	•	0	•	0	0	•	•	0	10.4	11.6	0.2	0.1	0.2	0.1	69.4	10.0

# Table 28

Deinking agent No. Component (a) Invention 1 C<sub>17</sub>H<sub>35</sub>COO(EO)<sub>18</sub>(PO)<sub>9</sub>H products 2 C<sub>11</sub>H<sub>23</sub>COO(EO)<sub>20</sub>(BO)<sub>7</sub>H 3 C<sub>17</sub>H<sub>33</sub>COO(PO)<sub>8</sub> (EO)<sub>8</sub>H C<sub>17</sub>H<sub>35</sub>COO(EO)<sub>90</sub>(PO)<sub>67</sub>H 4 5 C<sub>15</sub>H<sub>31</sub>COO(EO)<sub>50</sub>H 6 C<sub>17</sub> H<sub>35</sub> COO(EO)<sub>20</sub> (PO)<sub>20</sub> H 7  $C_{21}H_{43}COO(EO)_{30}(BO)_{10}H$ 8 C<sub>17</sub>H<sub>33</sub>COO(PO)<sub>10</sub>(EO)<sub>20</sub>H 9 C<sub>11</sub>H<sub>23</sub>COO(EO)<sub>60</sub>H

Table 29

Deinking age	ent No.	Weight ratio (b)/(a)	
Invention	1	90/10	
products	. 2	85/15	
	3	80/20	
	4	75/25	
	5	70/30	
	6	65/35	
	7	60/40	
:	8	50/50	
•	9	40/60	

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Table 30

Deinking agent No.		Qualities of deinked pulp		
		whiteness (%)	b value (%)	unliberated ink no.
Invention	1	55.4	10.0	6
products	2	55.3	10.2	5
	3	55.4	9.95	7
	4	55.5	10.1	6
	5	55.8	10.1	6
	6	56.5	10.3	4
*	7	55.7	10.2	5
	8	55.5	10.1	6
	9	55.4	9.98	7

#### Example 11

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In this Example, a deinking agent was added in portions in the pulping and chemical mixing stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.1% (based on the starting material) of each of the deinking agents listed in Tables 31 to 33 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Tables 31 to 33 were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30 °C for 10 minutes. During the flotation process, 0.4% (based on the starting material) of CaCl2 was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 31 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 32 shows the component (a) and Table 33 shows the weight ratio of the components (b) to (a). Further, Table 34 shows the deinking performances.

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Table 31

Table 32

Deinking age	ent No.	Component (a)
Invention	19	C <sub>11</sub> H <sub>23</sub> COO(EO) <sub>20</sub> (PO) <sub>10</sub> H
products	20	C <sub>17</sub> H <sub>35</sub> COO(PO) <sub>10</sub> (EO) <sub>10</sub> H
r .	21	C <sub>15</sub> H <sub>31</sub> COO(EO) <sub>12</sub> (BO) <sub>6</sub> H
	22	C <sub>17</sub> H <sub>33</sub> COO(EO) <sub>50</sub> H
	23	C21H43COO(EO)30(BO)10H
	24	C7H15COO(EO)70(PO)40H

Table 33

Deinking ag	ent No.	Weight ratio (b)/(a)
Invention	19	90/10
products	20	80/20
	21	70/30
• •	22	60/40
	23	50/50
	24	40/60

Table 34

Deinking ag	ent No.	Qualit	ies, of deinked p	oulp
		whiteness (%)	b value (%)	unliberated ink no.
Invention	19	55.9	10.1	5
products	20	56.4	10.3	4
	21	55.5	9.96	6
•	22	55.8	10.1	5
•	23	55.7	10.0	5
	24	55.7	10.21	4

## Example 12

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In this Example, a deinking agent was added in portions in the pulping and kneading stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.3% (based on the starting material) of each of the deinking agents listed in Tables 35 to 37 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3. 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Tables 35 to 37 were added thereto. After

adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 10° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 35 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 36 shows the component (a) and Table 37 shows the weight ratio of the components (b) to (a). Further, Table 38 shows the deinking performances.

The components (b) listed in Table 35 were cod, sardine, saury and mackerel oil fatty acids (No. 30 to No. 33) and the one of No. 34 was a mixture of the components (b) of No. 30 and No. 31 at a weight ratio of 50/60. Those of No. 35 to No. 36 were prepared by varying the iodine value (IV) of the one of No. 30.

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		Average C Todine atom No. value	Indias value						ž	y sold	og i bo	fatty seld earbon No. composition	a) sod	ion (vt.1)	=						Content of
//	. <u>F</u>		(17)	r.	່ນ	C <sub>t</sub> ,	l <sup>1</sup> O	n,	C,	C,	C,1	C,	C,	ď	C.	3	5	3	3	ئ	tatty acids
	30	10.5	0.6	0	0	•	0	0	0	1.9	0	36.9	•	19.5	•	17.0	•	23.9	٥	۰	41.7
products	n	10.5	0.1	0	0	•	•	0	0	6.5	0	23.0	0	27.6	0	20.2	•	19.1	0	3.1	40.9
	32	19.0	0.6	•	0	0	0	0	•	6.9	0	19.0	۰	23.0	0	22.7	0	27.5	0	6.0	51.1
	33	10.0	0.8	0	•	•	0	۰	•	6.0	•	22.7	0	23.6	•	20.0	•	26.7	•	٥	47.5
	×	10.5	0.1	•	•	0	0	0	0	4.2	0	31.0	•	23.6	0	19.0	0	21.5	0	0.8	41.3
	35	10.5	25.4	0	•	0	•	9	•	1.9	0	36.9	. 0	19.9	0	17.0	0	83.9	•	٥	41.7
	ž				•	•	•	•	•	•	,	• **	•	•	•	• • •	•	•	•	ľ	

Table 36

Deinking ag	ent No.	Component (a)
Invention	30	C <sub>17</sub> H <sub>35</sub> COO(EO) <sub>20</sub> (PO) <sub>10</sub> H
products	31	C <sub>11</sub> H <sub>23</sub> COO(PO) <sub>12</sub> (EO) <sub>12</sub> H
•	32	C <sub>17</sub> H <sub>33</sub> COO(EO) <sub>30</sub> (BO) <sub>15</sub> H
	33	C <sub>15</sub> H <sub>31</sub> COO(EO) <sub>60</sub> H
	34	C21H43COO(EO)10(PO)10H
	35	C <sub>17</sub> H <sub>35</sub> COO(EO) <sub>20</sub> (PO) <sub>10</sub> H
	36	C <sub>17</sub> H <sub>35</sub> COO(EO) <sub>20</sub> (PO) <sub>10</sub> H

Table 37

Deinking ag	ent No.	Weight ratio (b)/(a)
Invention	30	30/20
products	31	70/30
	32	60/40
	33	50/50
	34	40/60
·	35	80/20
	36	80/20

Table 38

Deinking ag	ent No.	Qualit	ies of deinked p	ulp
		whiteness (%)	b value (%)	unliberated ink no.
Invention	30	57.6	10.8	2
products	· 31	57.5	10.6	3
	32	57.6	10.5	3
	33	57.5	10.2	4
	34	57.7	10.8	2
	35	57.6	10.5	3
	36	57.5	10.1	4

## Example 13

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In this Example, a deinking agent was added in portions in the pulping stage and before the flotation stage.

Recovered waste newspapers were cut into pieces ( $2 \times 5$  cm) and a given amount thereof was fed into a bench disintegrator. Then water. 0.2% (based on the starting material) of caustic soda and 0.2% (based

on the starting material) of each of the deinking agents listed in Tables 39 to 41 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3 and 3.5% (based on the starting material) of 30% hydrogen peroxide were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. Then 0.3% (based on the starting material) of each of the deinking agents listed in Tables 39 to 41 was added to the obtained pulp slurry. After diluting with water so as to give a pulp concentration of 1%, the slurry was subjected to flotation at 30°C for 10 minutes. After the completion of the flotation, the pulp slurry was concentrated with a 60-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 40° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 39 shows the average carbon atom number of the component (b), the iodine value, the fatty acid composition and the content of fatty acids having 20 to 24 carbon atoms of each of the deinking agents. Table 40 shows the component (a) and Table 41 shows the weight ratio of the components (b) to (a). Further, Table 42 shows the deinking performances.

In Table 39, all of the fatty acids contained in the components (b) of the deinking agents were in the form of sodium salts. Each of the deinking agents No. 43 to No. 46 is prepared by blending individual sodium salts of the fatty acids with each other in such a manner as to give a composition of a specified carbon atom number.

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		Average C	lodine						Fatt	y sold	cerbor	Fatty sold carbon No. composition (wt.1)	positi	los (ve	١٠.						Content of
//	Æ		(14)	វ	°	C, C, C, C,	j.	c <sub>1</sub> ,	15	C,	C <sub>15</sub>	כו כו כיי כיי כיי כיי כיי כיי כיי כיי כי	C,	C,	<b>1</b> 5	C,n	ű	c,,	C,1	ų.	(atty acids
	69	19.0	1.5		•	0 0 0	٥	0	۰	1.0	•	0 7.0 0 14.0 0 17.4 0 10.1 0 20.3 0 22.4	۰	17.4	•	19.1	•	20.3	0	22.4	60.0
Invention products	:	18.0	3.3	•	•	•	۰	10.2	•	\$.2	•	0 0 0 10.2 0 5.2 0 0	•	60.3	0	9.0	•	0 60.3 0 8.0 0 16.3 0	0	0	24.3
	\$	17.7	6,9	0,9 4.2 0 10.3 0	0	10.3	•	1.9	•	3.9	•	8.9 0 6.4 0 8.8 0 10.2 0 19.8 0 18.3 0 13.1	•	10.2	•	19.0	•	11.3	0	13.1	51.2
	*	18.9	3.6 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	0	0	•	•	.0	۰	0	•	16.9	•	4.3	0	19.8	•	20.9	0	0.1	40.0

Table 40

Table 41

Deinking agent No. Weight ratio (b)/(a)

Invention 43 90/10

products 44 80/20

45 70/30

46 60/40

Table 42

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Deinking ag	ent No.	Qualit	ies of deinked p	ulp
		whiteness (%)	b value (%)	unliberated ink no.
Invention	43	57.5	10.8	, 2
products	44	57.6	10.8	2
	45	57.3	10.5	3
	46	57.2	10.5	3

Ŀ					Ì															
	Average C	Velue			٠,			Pat	Patty sold	carbon	ž	composition		(we.1)				·	Γ	Content o
6		(110)	5	J	J	Ĵ	<b>"</b> 5	15	75	1,5	ď	ئ	Ĵ	Ĵ	3	ð	J	3	ٿ	fatty sold
4	12.8	0.7	0.2	0.04	21.1	90.0	5.6	0.03	3	90.0	-	0.0	-	0.03	3	ş	3	8	[	
•	13.1	2.8	16.1	0.03	0.1	10.0	10.5	0.01	200	•	.:	8	3	0.0	5	8	3	ě		
٠	14.5	9.9	9.7	0.03	2.3	10.0	10.5	0.03	1.15	L°	23.9	0.0	9.5	0.0	=	8	7	8	3	12.5
٥	15.6	1.7	5.6	0.01	0.5	0.01	3.7	0.02	3.5	0.02	3	9.0	-	0.03	3	8	-	8	3	12.5
•	16.0	2.1	3.2	۰	1.0	0	2.1	•	6.3	•	16.7	90.0		:	+	80.00	-	8	3	3
٠	13.4	3.5	1.2		3.2	0	2.1	0	11.4	0	10.4	9.0	2.2	9.0	3	3	+-	8	3	13.6
٥	10.2	1.9	3.6	0	0	0	0.1	0	=	•	3	0.03	ž.š.	8:	-	8	ä	8	3	ş
-	10.9	3.4	9.3	0	9.2	0	0.3	•	•.5	•	3.5	.03	÷.	.0	-	3	=	8	3	2.5
-	19.3	;	9:	٥	9.1	•	0.3	0	8.0	•	34.1	•	3.2	3	3	3	ž	3	3	2.5
-	20.4	1:0	°	۰	•	•	0.1	•	1.0	•	19.4	•	17.6	•	::	<del>                                     </del>	┺	3	2	2
_	21.2	3:6	۰	•	0	•	•	0	1.4	•	2.9	•	32.0		3	<del>-</del>	₩	3	ä	3
	2.5	7.9	•	۰	۰	۰	۰	•	0	•	0	•	10.4	11.6	0.2	1.0	0.2	7.	:	. ŏ
=	12.0	1.7	0.2	8	21.1	90.0	3.63	90.0	•	70.0	6.0	70.0	:	:	:	1	:	1	:	

Table 43 (cont'd)

7 O C	olds ()			•										,
Content of	(vc. 1)	12.5	12.0	16.5	37.3	62.0	70.0	41.7	40.9	51.1	47.5	=	=	41.7
	c <sub>j</sub> ,	0.1	0.1	0.1	3.7	23.5	1.69	۰	1:0	0.9	•	•	۰	•
	Cn	0.03	0.08	0.03	0.03	0.1	0.1	۰	۰	۰	٥	۰	۰	0
	C <sub>n</sub>	7.5	9.1	10.1	10.1	21.2	0.3	23.9	13.1	27.5	26.7	21.9	23.5	23.9
	J.	0.05	0.05	0.08	0.05	0.1	0.1	0	0	•	۰	۰	•	0
	S.	4.6	5.1	5.6	15.4	17.9	9.2	17.6	20.2	22.7	20.6	19.0	17.0	17.0
.11	ď	0.02	0.03	0.04	0.05	0	11.6	0	٥	0	۰	•	•	•
ion (ve	775	0.5	9.0	34.1	47.0	17.6	10.4	19.5	27.6	23.0	23.6	23.6	19.5	19.8
mposit	ڻ	0.01	10.0	90.0	0.08	0	•	•	0	•	•	•	0	•
fatty acid carbon No. composition (wt.1)	Clt	22.9	44.0	36.7	14.6	19.4	0	36.9	25.0	19.0	22.7	31.0	36.9	36.9
carbon	ď	0.03	0.02	•	۰	•	0	0	•	•	•	0	•	•
y acid	ڙ	41.5	29.6	:	:	:	•	1.9	6.5	6.9	6.0	63	=	3
I SE	Ĵ	0.03	0.03	•	•	•	۰	۰	•	•	•	•	•	•
	ניי	10.5	3.3	2.1	0.3	1:0	•	۰	۰	۰	•	•	•	•
ŀ	ניו	10.0	10.0	۰	•	•	۰	۰	•	•	۰	•	۰	•
	3	2.3	0.5	1.0	6.2	•	٥	۰	•	•	•	۰	•	۰
	ਹ	0.0	9.0	•	۶	•	•	•	•	·	·	ŀ	ŀ	•
	ۍ	:	3	3	3	•	•	ŀ	-	ŀ	ŀ	ŀ	•	ŀ
lodine	Ē	2	6.61	26.6	3	3.15	3	3	6.7	3	3	6	23.5	
Average C		16.5	2.5	2	6.01	2 8	2.5	5	2	19.0		3	\$ 11	
	ह	2	۰	•			-	Ŀ	Ŀ	,	-		•	ŀ

Example 14

In this Example, a deinking agent was added as a whole in the pulping stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.8% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.5% (based on the starting material) of each of the deinking agents listed in Table 43-44

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were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydraeor until the pulp concentration reached 23% and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegraeor for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Further, the foam volume at the flotation was measured as an indicator of the operation stability in the flotation stage. A foam volume ranging from 200 to 350 ml indicates a good stability. When the foam volume is outside the above range, foaming troubles might occur.

Table 44 further shows the component (b), the composition of the component (a) and the weight ratio of the components (b) to (a) of each of the deinking agents used in the test as well as the deinking performance thereof.

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		Tosa vol.	910	308	300	308	SOE	. 500	200	300	300	295	270	200
		Chhliberated Ink No.	\$	s	s	4	•	•	9	3	•	9	7	7
		en co	9.91	96.6	10.0	9.90	9.98	10.1	10.4	10.6	10.2	10.2	9.99	10.2
		Whiteness (1)	85.0	55.1	. 88.7	55.5	\$3.9	55.4	55.9	56.4	55.0	55.9	55.2	\$6.1
	1 1 1 1 1 1	(b)/(d) (wt. ratio)	10/30	78/22	21/00	76/24	74/26	98/36	90/10	. 05/15	81/18	61/20	93/0	90/10
Table 44	Γ	×	Z.	Me	. W.	PL.	H.	E.	*	×	1	, MA	MM,	, Mar
Tab		٩	2.0	2.0	2.0	4.5	1.0	8.0	3.0	2.0	2.4	3.0	1.2	3.8
	component (3): RO(AD),50,H	Of	E0/P0(1/1)	20	٤	E0/B0(1/1)	82	22	83	8	82	E0/F0(2/1)	, OZ	8
	component (	starting alcohol	1-octanol	1-octanol	1-octanol	1-oct anol	2-octanol	2-oct anol	oleyl alcohol	cyclononanol	2-octen-1-01	linolenyl alcohol	eleidyl. elcobol	elecatearyl alcohol
	component (b)	fatty acid	4	•	υ	,		•	9		1	C.	×	•
		. Ho.	-	~	•	3	~	•	•	-	•	10	:	13
		Deinking agent Ho.	•	Invention products										

The values given in the perentheses are molar ratios.

Alkylene oxides were all added at random.

Example 15

In this Example, a deinking agent was added in portions in the pulping and chemical mixing stages. Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.1% (based

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on the starting material) of each of the deinking agents listed in Tables 43 and 45 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Table 45 were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the, bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.4% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification). Further, the foam volume in the flotation stage was measured similar to Example 14.

Table 45 shows the component (b), the composition of the component (a) and the weight ratio of the components (b) to (a) of each of the deinking agents used in the test as well as the deinking performance thereof.

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The calues given in the parentheses are molar ratios.

Moter Alkylene oxides were all added at random.

73/27	4.6 MM, 73/27 85.3	E0/P0(2/1) ~4.2 K 72/20 55.4 10.2 6	20/9019/1) 2.9 K 74/26 85.5 10.2 S	E0/P013/11 1.2 K 80/20 55.6 10.2 4	E0 1.2 K 02/16 55.3 10.1 3	EO 1.8 K 90/10 85.3 10.2 4	EO 2.1 No 69/12 55.4 10.3 \$	AD B (vt. ratio) (1) (1) Ink Mo.	antenness d
73/27	73/27	72/20	14/26	80/20	62/16	90/10	69/12	-	-
+	╄	_		$\dashv$		-	┥		
+	╁	۲	Н	Н				9	1. RO (AO) .BO.H
octyl phenol	and where i	limeteri elcohol	11-dodecen-1-ol	2-tetracosanol	2-nonadecanol	2-henadecanol	2-pent adecanol	statting slephol	component (
•		•	۰	•	°	=	¥	fatty acid	component (b)
•	<u>.</u> †	:	:	:	:	=	2.0	Deinking agent No.	

Example 16

In this Example, a deinking agent was added in portions in the pulping and kneading stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.3% (based on the starting material) of each,of the deinking agents listed in Table 46 were added thereto. After

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disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 22%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based-on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Table 46 were added thereto. After adjusting the pulp concentration to 23% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 10° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification). Further, the foam volume was measured in the flotation stage similar to Example 14.

Table 46 shows the component (b), the composition of the component (a) and the weight ratio of the components (b) to (a) of each of the tested deinking agents and the deinking performance thereof.

The components (a) listed in Table 9 were cod, sardine, saury and mackerel oil fatty acids (No. 75 - No. 78); T, U, V and W) and the one of No. 79 (X) was a mixture of the components (b) of No. 75 (T) and No. 76 (U) at a weight ratio of 50/50. Those of No. 80 were prepared by varying the iodine value (IV) of the one of No. 75 (T).

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\$6.3 \$6.5

		(q) component		component ( G ): 80 (A0) ,50 M			(b)/(a)				•
Deinking egent No.		fatty acid	statting alcohol	AO	£	×	(wt. satio)	Internation (1)	(1)	MALTENGES D'ALUS UMALIDETATES FOS	2
	74	4	nonylphenol	E0/P0(1/1) 1.2 Na	1.2	7.	61/19	86.4 10.3	10.3	1	
Invention products	7.5	, 0	1-heneicosanol	£0	2.4 No	Ka	12/28	36.3 10.6	10.6	. 1	
	3,6	^	2-tricosanol	93	4.3 10	T.	12/17	86.0 10.5	10.5	0	

inte: Alkylene oxides were all added at random. The values given in the perenthages are molar ratios.

E0/P0(2.5/1)

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2-tetracosanol

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## 55 Examples 17

In each of these Examples, a deinking agent was added in portions in the pulping and chemical mixing stages.

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Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and a given amount of each of the deinking agents listed in Tables 47 and/or 2 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and a given amount of each of the deinking agents listed in Tables 47 and/or 48 were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the notation process, 0.4% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 49 shows the deinking performances achieved by various deinking methods. Each of the highly fatty acid mixtures  $\alpha$  to  $\delta$  listed in Table 48 is prepared by blending individual fatty acids with each other in such a manner as to give a composition of a specified carbon atom number.

Table 47

No.	Surfactant: (a-1) to (a-4)	Mode of AO addition
0	(a-1) C <sub>18</sub> H <sub>33</sub> O(PO) <sub>10</sub> (EO) <sub>10</sub> H	block
2	(a-1) C <sub>16</sub> H <sub>33</sub> O(PO) <sub>12</sub> (EO) <sub>6</sub> H	block
3	(a-2) C <sub>11</sub> H <sub>23</sub> COO (EO) 20 (PO) 10H	block
4	(a-3) CH <sub>2</sub> =CH (CH <sub>2</sub> ) <sub>10</sub> O (EO) <sub>2.4</sub> (PO) <sub>0.5</sub> SO <sub>3</sub> K	random
5	(a-4) hardened palm oil (IV = 1.2)/ diglycerol mixture (1/2.6 by mol) (PO) <sub>37.2</sub>	random

In the above table, AO means an alkylene oxide and the same will apply hereinafter.

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		Content of	fatty acids (vt.N)	37.3	62.0	10.9	70.0
				3.7	23.5	3	5.6
			C <sub>1</sub> ,	0.03	9.1	0.03	0.1
			CB	1.5	21.2	6.2	0.3
			ű	0.05	0.1	0.03	0.1
			2	13.4	17.9	5:	6.2
		(4)	C,	0.03	0	0.03	11.6
Į	nt (b)	on lut.	ů	47.0	17.6	3.5	::
	compon	mposti	ئ	0.08	•	9.0	•
	Higher fatty sold mixture: component (b)	fatty sold carbon No. compostion (wt.1)	t) (15 (16 (17 (17 (18 (18 (18 (18 (18 (18 (18 (18 (18 (18	3.2 0 0.2 0 0.3 0 0.3 0 14.6 0.08 47.0 0.08 18.4 0.08 18.1 0.09 3.7	0 0.1 0 0.1 0 19.4 0 17.6 0 17.9 0.1 21.2 0.1 23.5	.2 0.04 21.1 0.06 63.6 0.05 0.2 0.06 0.2 0.04 3.5 0.05 4.5 0.05 6.2 0.05 0.1	0 0 10.4 11.6 0.2 0.1 0.2 0.1 69.4
	acid mi	carbor	Cu,	0	•	90.0	0
	Catty	ty actd	c,	0.3	0.1	0.2	•
	Higher	Tat		0	•	0.03	0
			J.	0.3	9.1	13.6	0
			ű	٥	۰	90.0	0
			Ĵ	9.2	0	1.15	9
			ľ	0	٥	0.04	9
			ű	9.3	•	2.0	٩
		Iodine	ē	9.50	31.5	1.1	5
		Average	etom No.	16.9	20.4	12.0	3.3
		-	<u> </u>	8	-	-	4

Table 48

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	Stage and a	tage and amount of addition of deinking agent	inking agent				
Ex. No.	deinking ac start	einking agent (wt.% based on starting material)	deinking agent	Whiteness	b value	Unliberated	
	pulping stage (I)	chemical mixing stage (II)	(wt. ratio)				
-	Φ (0.2)	α (0.2)	50/50	56.1	10.6	. 3	
2	α (0.2)	Φ (0.2)	50/50	54.1	90.6	8	
m	Ø (0.1)	β (0.2)	33/67	56.0	10.7	3	
4	Ø (0.045) β (0.055)	Ø (0.09) β (0.11)	45/55	55.7	10.4	9	
5	<b>(0.1)</b>	γ (0.2)	33/67	56.1	10.7	3	
9	γ (0.2)	(0·1)	33/67	54.2	9.05	6	
7	(0.01) 7 (0.09)	. ① (0.02) . 7 (0.18)	10/90	55.9	10.1	w	
8	(0.126)	α (0.324)	28/72	56.4	10.9	2	
6	<b>Φ</b> (0.056) α (0.144)	<b>Φ</b> (0.07) α (0.18)	28/72	55.5	10.2	w	
10	© (0.1)	8 (0.2)	33/67	56.3	10.9	3	
1	\$ (0.1)	<b>©</b> (0.2)	67/33	.54.1	9.05	6	
12	δ (0.027) δ (0.073)	© (0.054) 6 (0.146)	27/73	56.3	10.3	+	

## 55 Examples 18

In each of these Examples, a deinking agent was added in portions in the pulping and kneading stages. Recovered waste newspapers were cut into pieces  $(2 \times 5 \text{ cm})$  and a given amount thereof was fed into

a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and a given amount of each of the deinking agents listed in Tables 50 and/or 51 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Tables 50 and/or 51 were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 10° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 52 shows the deinking performances achieved by various deinking methods. The higher fatty acids contained in the deinking agents listed in Table 51 were hardened fatty acids of sardine, saury and mackerel oils (Nos.  $\epsilon$  to  $\eta$ ) and stearic acid and sardine oil fatty acids (IV = 175) (Nos.  $\theta$  and 1).

Table 50

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monthly and the second	No.	Surfactant: (a-1) to (a-4)	Mode of AO addition
30	6	(a-1) C <sub>18</sub> H <sub>37</sub> O(EO) <sub>70</sub> (PO) <sub>30</sub> H	random
	Ø	(a-2) C <sub>17</sub> H <sub>33</sub> COO (EO) <sub>30</sub> (BO) <sub>15</sub> H	random
	8	(a-3) C <sub>22</sub> H <sub>45</sub> (CH <sub>3</sub> ) CHO (EO) 3.2SO3Na	-
35	9	(a-4) hardened fish oil (IV = 0.8)/ ethylene glycol mixture (1/1.6 by mol) (EO) <sub>39.9</sub> (PO) <sub>13.3</sub>	block
<b>40</b>	0	<pre>(a-4) hardened beef tallow (IV = 2.0)/ glycerol mixture (1/0.6 by mol) (EO)<sub>52</sub>(PO)<sub>29</sub></pre>	block

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Table 51

	ent of	fatty solds	40.9	51.1	1.5		6.9
	Ş		ľ	Ĩ	ľ		•
		نّ	=	:	۰	0	1.6
		3	۰	۰	۰	0	0
		3	19.1	27.5	26.7	0	19.1
		5	٥	۰	•	0	0
		Ca Ca Ca	20.2	22.7	20.6	0	20.2 0
	3	C <sub>10</sub> C <sub>10</sub>	0	0	0	0	0.
ent (b	<b>8</b>		27.6	23.0	23.0	100	27.6 .0
Compon	ompost (	C,	0	0	0	0	0
Migher fatty acid mixture: component (b)	fatty acid earbon No. compostion (vt.1)	r,	25.0	19.0	12.7	0	25.0
scld .	1 carbo	C.19	0	0	. 0	0	0
fatty	ty sole	C,1 C,1	6.3	6.9	6.0	0	6.5
Higher	Fat	619 C11 C13 C19	. 0	•	•	•	0
		C,	0	0	0	0	0
	,	C,	•	۰	۰	۰	•
		33	٥	•	۰	۰	•
		2	۰	۰	۰	۰	•
		ů	۰	۰	é	۰	۰
	lodine	(TV)	6.3	9.3	9.3	0.3	175.0
	Average	atom Mo.	18.5	19.0	19.9	10.0	10.5
	.98		-	4	.5	•	-

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<b>30</b>			
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	Stage and a	tage and amount of addition of deinking agent	inking agent			
Ex. No.	deinking ag start	einking agent (wt.% based on starting material)	deinking agent	Whiteness (%)	b value	Unliberated.
	pulping stage (I)	chemical mixing stage (II)	(we. raelo)		•	
1	<b>6</b> (0.15)	e (0.35)	30/70	58.8	11.4	0
. 2	(0.045) c (0.105)	(0.105) (0.245)	30/70	57.6	10.7	4
3	Ø (0.2)	ζ (0.3)	• 09/0	58.2	11.3	1
4	ζ (0.2)	Ø (0.3)	09/01	55.3	10.1	9
2	Ø (0.08) \$ (0.12)	Ø (0.12) \$ (0.18)	09/04	. 57.1	10.5	v
. 9	(0.125)	ŋ (0.375)	25/75	58.1	11.3	0
7	இ (0.031) ்ர (0.094)	(0.094) n (0.281)	25/75	56.4	10.7	s
æ	@ (0.225)	ຖ (0.275)	45/55	58.2	11.2	0
6	(0.100) η (0.125)	(0.125) n (0.15)	43/55	56.5	10.7	'n
10	(0.225)	0 (0.275)	45/55	52.6	7.34	16
=	(0.15)	£ (0.35)	30/70	59.2	11.3	2
. 12	@ (0.15)	(0.35)	30/70	52.0	7.22	18
13	(0.06) (0.14)	(0.09) ( (0.21)	30/70	52.1	7.24	19

Example 19

In each of these Example, a deinking agent was added in portions in the pulping stage and before the flotation stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into

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a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and a given amount of each of the deinking agents listed in Tables 53 and/or 54 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 22%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3 and 3.5% (based on the starting material) of 30% hydrogen peroxide were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. Then a given amount of each of the deinking agents listed in Tables 53 and/or 54 was added to the obtained pulp slurry. After diluting with water so as to give a pulp concentration of 1%, the slurry was subjected to flotation at 30°C for 10 minutes. After the completion of the flotation, the pulp slurry was concentrated with a 60-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 40° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 55 shows the deinking performances of various deinking methods. In Table 54, all of the fatty acids contained in the deinking agents were in the form of sodium salts. Each of the higher fatty acid mixture x to  $\pi$  is prepared by blending individual sodium salts of the fatty acids with each other in such a manner as to give a composition of a specified carbon atom number.

Table 53

98%		<u> </u>
No.	Surfactant: (a-1) to (a-4)	Mode of AO addition
0	(a-1) C <sub>12</sub> H <sub>25</sub> O(EO) <sub>10</sub> (PO) <sub>5</sub> H	random
0	(a-1) C,H1,-(O)O(EO), (PO),H	random
(3)	C <sub>6</sub> H <sub>13</sub> O (EO) <sub>10</sub> (PO) <sub>5</sub> H	random.
<b>(3</b> )	C <sub>28</sub> H <sub>57</sub> O (EO) <sub>5</sub> (PO) <sub>5</sub> H	random
(3)	C <sub>18</sub> H <sub>37</sub> O (EO) <sub>150</sub> (PO) <sub>80</sub> H	random
<b>6</b>	(a-2) C <sub>15</sub> H <sub>31</sub> COO (CO) 10 (BO) 5H	· block
0	C <sub>5</sub> H <sub>11</sub> COO (EO) <sub>18</sub> (PO) <sub>9</sub> H	block
13	C <sub>17</sub> H <sub>35</sub> COO (EO) <sub>2</sub> (PO) <sub>1</sub> H	block
<b>(9</b>	C <sub>27</sub> H <sub>55</sub> COO (EO) <sub>10</sub> (PO) <sub>10</sub> H	block
89	C <sub>17</sub> H <sub>35</sub> COO (EO) <sub>200</sub> ( (PO) <sub>150</sub> H	block
<b>છ</b>	(a-3) C <sub>8</sub> H <sub>17</sub> O(EO) <sub>1</sub> (PO) <sub>1</sub> SO <sub>3</sub> Na	random
8	(a-4) coconut oil/pentaerythritol mixture (1/0.48 by mol) (EO) 35 (PO) 19.4	random

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Table 54

								Higher	fatty	acld m	Higher fatty acid mixture: component (b)	COMPON	1							
	Average	lodine						Ta.	ty act	d carbo	Fatty seld earbon No. compostion (wt.1)	ompost L	on (wt.	3.						Content of
<u>ė</u>	atom No.	200	J	៤	ن	3	3	ß	ני	<b>3</b>	C <sub>1</sub> 0	a <sup>1</sup> 2	Ç,	J.	C <sub>B</sub>	ű	C.	C,	, <sub>1</sub> ,	fatty acids (vt.1)
	13.0	:	•	۰	۰	•	•	۰	7.0	0	14.0	•	17.4	•	10.1	٥	Š.	۰	22.4	60.0
-	2,2	:	3	•	10.3	.•	:	۰	3	0	1.1	0	10.2	•	19.0	0	11.3	۰	13.1	51.2
	9 91		•	•	•	•	10.2	۰	5.2	•	۰	0	60.3	0	0.0	•	16.3	0	•	24.3
1.		,	.0	8.6	2.1	90.0	3	0.2 0.04 21.1 0.06 63.6 0.05		0.2 0.06	0.2	0.2 0.04	-:	3.8 0.05 4.5 0.03	4.5	0.03	5.9 0.03	9.0	0.1	9.6
-	2	-	1	•	۰	۰	٥	۰	۰	0	•	0		•	٥	•	۰	•	10.9	10.9
-	13.2	:	5.0	0.02	:	9.0	±.:	0.1 0.01 27.7 0.04 45.7 0.01	45.7	0.01		0.4 0.02	0.5	-	5:5	3.5 0.05	3.5	8.0 0.08	0.1	9.8
Ŀ			٩	٩	9.5	۰	•	۰	18.7	÷ 0	12.0	0	0.3	0.1	23.4	0.08	0.2 0.1 29.4 0.05 35.4 0.05	0.03	5.0	70.7

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Table 55

	Stage and a	age and amount of addition of deinking agent	inking agent		·	
EX.	deinking ag	inking agent (wt.% based on starting material)	deinking agent	Whiteness (%)	b value	Unliberated ink No.
·	pulping stage (I)	chemical mixing stage (II)	(b)/(a) (wt. ratio)	•		
1	(0.2)	(c·o) (D	09/01	59.2	11.8	0
2	(0.02) × (0.18)	Q (0.03) × (0.27)	10/90	87.8	10.7	2
3	·@ (0.08) K (0.12)	(0.12) K (0.18)	40/60	56.4	10.4	5
Þ	(O (0.15)	(B (0.35)	30/70	58.9	11.5	1
5		(β (0.09) λ (0.21)	30/70	57.6	10.8	2
9	(O (0.2)	μ (0.3)	40/60	59.0	11.6	0
2	(0.08) µ (0.12)	<b>②</b> (0.12) μ (0.18)	40/60	55.4	10.2	v
8	(0.15)	λ (0.35)	30/70	59.1	11.6	0
6	λ (0.35)	(§ (0.15)	30/70	55.5	11.0	3
10	(0.04)	v (0.36)	10/90	58.7	11.4	
11	Q (0.02)	② (0.02) v (0.18)	10/90	55.1	10.1	so.

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<b>20</b>		Table 55 (cont'd)
25	· ·	ble 55
		Ta
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<b>45</b> ·	:	

	Stage and a	Stage and amount of addition of deinking agent	inking agent			
Ex. No.	deinking ag	einking agent (wt.% based on starting material)	deinking agent	Whiteness (%)	b value	Unliberated ink No.
	pulping stage (I)	chemical mixing stage (II)	(D)/(a) (wt. ratio)			
12	Ø (0.1)	v (0.4)	20/80	58.9	11.5	ĿΩ
13	(%) (0.02) v (0.08)	② (0.08) v (0.32)	20/80	55.3	10.2	, K
14	.O (0.2)	μ (0.3)	40/60	59.0	11.0	1
15	. ⊕ (0.2)	μ (0.3)	40/60	54.8	9.99	10
16	(0.04) µ (0.16)	Θ (0.06) μ (0.24)	20/80	51.4	7.58	15
17	μ (0.2)	(O (0.3)	60/40	50.5	7.55	11
18	11 (0.2)	μ (0.3)	40/60	59.9	11.3	1
19	(0.2)	μ (0.3)	40/60	53.8	9.30	13
20	(0.2)	μ (0.3)	40/60	53.8	8.91	10
21	(D (0.2)	μ (0.3)	40/60	54.7	9.90	12
22	Ø (0.1)	K (0.4)	20/80	59.3	11.0	2
23	(1.0)	£ (0.4)	20/80	50.3	6.63	30
24	(O (0.1)	0 (0.4)	20/80	49.9	6.42	29
25	Ø (0.1)	т (0.4)	20/80	49.8	6.42	29
		7.				

As the above Examples show, a deinked pulp having a high whiteness and a high b value and contaminated with little unliberated ink can be obtained by adding one or more surfactants selected from among the compounds represented by the general formulae (a-1) to (a-3) and a reaction product obtained by adding an alkylene oxide to a mixture of a natural fat with a polyhydric alcohol (a-4) exclusively in the disintegration (pulping) stage of waste paper and adding a mixture containing higher fatty acid(s) having 8

to 24 carbon atoms or salt(s) thereof, wherein the average carbon atom number of the fatty acids in the mixture ranges from 12.7 to 22.5, the content of higher fatty acid(s) having 20 to 24 carbon atoms or salt(s) thereof ranges from 9.6 to 70.6% by weight and the iodine value (IV) is not more than 45, exclusively in the mixing and/or flotation stages following the pulping stage, preferably at a specific ratio.

	Average C	lodine				r		rate	plos ti	carbon	fatty sold carbon No. composition (wt.1)	) Teodi	lon (v	3						Content of
99		(1)	ຸ"ວ	t'o	C <sub>13</sub>	1º2	C <sub>1</sub> ,	3	ů	ئ	3	ۍ	نْ	ڻ	3	ű	3	ď	ű	fatty solds
٧	12.8	0.7	0.2	0.04	11.12	90.0	63.6	0.05	0.2	90.0	0.3	0.04	3.1	0.03	4.5	0.08	8.9	0.03	6.	9.6
-	13.1	2.0	16.1	0.03	0.1	0.01	40.8	10.0	10.0	0	0.3	10.0	0.4	0.08	6.1	0.03	6.9	0.05	0.1	11.0
U	11.5	0.0	9.7	0.03	2.3	10.0	10.5	0.02	41.5	0.02	22.9	10.0	0.9	0.03	1.1	0.05	1.5	0.05	0.1	12.5
٥	15.6	1.7	9.6	10.0	0.5	10.0	3.7	0.03	29.6	0.03	14.0	10.0	0.6	0.03	1.8	0.08	•	0.03	0.1	12.0
	16.0	2.1	3.2	0	7.0	0	1.1	0	0.3	0	36.7	90.0	34.1	0.04	9.6	0.05	10.7	0.08	0.1	16.5
_	17.4	3.5	1.2	0	3.2	0	2.1	0	12.4	0	10.4	0.04	\$2.0	90.0	8.0	0.05	11.4	0.03	0.5	17.0
0	10.2	1.9	3.0	0	•	0	0.1	0	0.1	0	0.1	0.07	74.8	0.03	5.3	0.03	13.4	0.03	1.1	30.8
Ξ	10.9	5.4	0.2	•	0.2	0	0.3	0	0.3	0	14.6	0.09	47.0	0.03	18.4	0.03	10.1	0.03	5.3	37.3
-	19.3	1.1	0.1	0	0.1	Đ	0.2	•	0.2	0	36.1	0	9.1	1.0	16.5	1.0	26.9	0.1	10.4	54.0
S.	20.4	1.6	0	0	0	0	0.1	0	0.1	0	19.4	0	17.6	0	17.9	0.1	21.2	0.1	23.5	62.0
×	21.2	7.6	0	0	•	0	0	0	0.1	0	2.9	0	32.9	0	4.2	0.1	20.6	0.1	39.1	64.1
	22.3	6.2	0	0	0	0	•	0	0	0	0		10.4	11.6	0.2	0.1	0.2	0.1	69.4	70.7
×	12.8	1.7	0.2	0.04	21.1	90.0	63.6	0.03	0.2	90.0	0.2	0.04	3.9	0.08	4.5	0.03	6.2	0.03	0.1	10.9

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Table 56 (cont'd)

12.0 0.0 23.5 : ŝ 29.5 Ĵ (M. ) 23.0 23.6 27.6 Ĵ ż 23.0 carbon Ĵ **p**cld .. j 0.03 Ĵ 3 6.7 22.3 36.5 : · · · 19.9 20.1

## ss Example 20

In this Example, a deinking agent was added as a whole in the pulping stage.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into

a bench disintegrator. Then water, 0.8% (based on the starting material) of caustic soda. 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.5% (based on the starting material) of each of the deinking agents listed in Table 57 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the pulp concentration reached 23% and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub>(Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with in image analyzer (100 x magnification).

Further, the foam volume at the flotation was measured as an indicator of the operation stability in the flotation stage. A foam volume ranging from 200 to 350 ml indicates a good stability. When the foam volume is outside the above range, foaming troubles might occur.

Table 57 further shows the component (b), the composition of the component (a) and the weight ratio of the components (b) to (a) of each of the deinking agents used in the test, while Table 58 shows the deinking performance thereof.

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Table 57

		Component (b)		Component (a)				10//10/
Deinking agent	Je J	- 77	natural fat	polyhydric sleebol	۵/۵	alkylene oxide	P	(of: ratio)
		MINCULO	(8)	(4)	(moier ratio)	type, molar ratio	mol no.	
	_	V	cocont oil	pentaerythritol	1/0.40	£0/P0(1.8/1.0)	54.4	10/30
Invention		•	beef tallow	1, 6-henenedlol	1/0.5	E0/P0(2.0/1.0)	14.6	\$9/65
		٥	soybeen oil	glycerol	1/0.6	E0/P0(2.2/1.0)	30.0	11/00
	-	٥	rapeseed oil	erytheitol	1/1.5	E0/P0(2.0/1.0)	43.2	66/34
-	_	3	cestor oil	pentserythritol	1/2	E0/P0(2.6/1.6)	93.0	34/36
	-		linseed oil	stachyose	1/1.2	E0/P0(2.1/1.0)	54.0	06/14
	-	5	palm of 1	mennitoi	1/0.0	2	18.9	\$0/50
-	-	=	fish oil	sorbitel	1/0.19	<b>04</b> .	19.0	68/18
	-	-	hardened beef taller (IV = 2.1)	srabitol	1/0.28	EQ/E0(4.2/1.0)	30.6	45/55
	2	,	hardened (1sh oil (1v = 0.9)	ethylane glycol	1/2.0	E0/P0(1.9/1.0)	13.0	62/38
	=	-	hardened coconut of 1 (IV = 1.6)	2-ethylbutane-1,2,3-triol	1/2.4	E0/P0(1.0/1.0)	27.0	32/40
	1	1	hardened paim oil (IV - 1.2)	sorbitol	1/2.5	E0/P0(0.25/1.0)	43.2	\$0/10
	1	•	coconut of 1	pentaerythritol	1/0.09	E0/P0(1.8/1.0)	\$4.4	10/30
	: =	•	esconst oil	penteerythritol	1/3.2	E0/P0(1.8/1.0)	54.2	10/30
	1	•	cocost oll	pentaerythritol	1/6.40	10/10/11/01	4.2	70/30

Mote: The altylene onide mol no. given in the above Table means the mol no. per mole of the matural (at (a) (the same will apply hereinafter).

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Table 57 (cont'd)

J	Component (s)		Component (b)				
Detaking agent	fatty sold mixture	natural for	-	e/s	elkylene oxide	8	(a) / (b) (vt. retio)
					type, molar ratio mol no.	mol no.	
16	*	fish oil	sorbitel	1/0.00	2	:	
products 17	×	tie dil	sorbitol	:	2 2		el/ce
		//eb of 1			2		13/13
			sorbitoi .	1/0.15	2	3.6	85/15

no. given in the above Table means the dol no. per mole of the natural fat (m) (the same will apply herelaafter).

Table 58

Deinking ag	jent No.	Whiteness (%)	b value (%)	Unliberated ink no.
Invention	1	55.5	9.70	6
products	2	55.4	9.81	7
	3	55.4	9.52	7
	4	55.3	10.0	7
	5	55.3	10.1	. 7
	6	55.2	10.0	. 8
	7	56.4	10.4	5
•	8	56.9	10.8	2
	9	56.5	10.5	5
	10	56.0	9.58	- 8
	11	55.9	9.97	6
	12	55.8	9.74	7
	13	54.6	9.58	10
	14	54.5	9.56	10
	15	54.7	9.49	11
	16	54.4	9.51	11
	17	54.6	9.53	10
	18	54.7	9.48	12

#### 35 Example 21

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In this Example, a deinking agent was added in portions in the pulping and chemical mixing stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the Starting material) of caustic soda and 0.1% (based on the starting material) of each of the deinking agents listed in Table 59 were added thereto. After disintegrating at a pulp concentration of 15% at 46°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reacted 23%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based on the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Table 59 were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.4% (based on the starting material) of CaCl2 was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 5° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (CA/MS = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained were measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyser (100 x magnification).

Table 59 shows the component (b), the composition of the component (a) and the weight ratio of the

components (b) to (a) of each of the deinking agents used in the test, while Table 60 shows the deinking performance thereof.

		Component (b)		Component (8)				
Desniting agent	,	fatty acid	yeu	polyhydric sleehol	d/o	alkylene oxide	qe.	(b)/(8)
	T		(5)	Tab Car	(MOLAF FALLO)	type, molar ratio	mol no.	
100000	=	*	beef tallow	sorbitol	1/0.1	E0/P0(1.0/1.0)	60.0	200
products	=	2	hardened bone oil (IV = 2.0)	propylene glycel	1/1.2	2	147.3	97/96
	=	٥	hardened beef tellow (IV - 0.6) trimethylene glycol	trimethylene glycol	1/0.5	2	9.0	81/20
	=	•	coconut of 1	butylene glycol	1/2.2	10.1/9.11004/03	3	\$0/30
	=	0	olive oil	1,1,1-trimethylotherane	1/1.5	E0/P0(0.5/1.0)	ä	*
	5		paim oil	1,2,6-hezametriol	1/2.4	.83	ž.,	62/39
	=	•	herdened palm oil	digiyeerol	1/2.6	2	37.2	נג/נג
	=	I	beef tallow	sorbitol	1/0.00	E0/P0(2.0/1.0)	69.0	66/33
	2	z	beef tallow	serbitol	1/3.1	E0/P0 (2.0/1.0)	60.0	68/32
	23	=	best tallow	sorbitol	1/0.1	EO/PO(2.0/1.0)	3.0	66/32

Table 60

Deinking ag	gent No.	Whiteness (%)	b value (%)	Unliberated ink no.
Invention	42	55.7	10.2	5
products	43	55.5	. 10.1	6
	. 44	55.6	10.1	6
	45	55.5	10.0	7.
	46	56.6	10.7	3
13.0	47	56.4	10.4	4
	48	56.3	10.3	4
	49	54.1	9.43	- 11
	50	54.6	9.38	13
	51	54.3	9.49	12

### Example 22

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In this Example, a deinking agent was added in portions in the pulping and kneading stages.

Recovered waste newspapers were cut into pieces (2 x 5 cm) and a given amount thereof was fed into a bench disintegrator. Then water, 0.2% (based on the starting material) of caustic soda and 0.3% (based on the starting material) of each of the deinking agents listed in Table 61 were added thereto. After disintegrating at a pulp concentration of 15% at 45°C for 12 minutes, the mixture was aged at 55°C for 120 minutes. Next, the mixture was dehydrated with a high-speed dehydrator until the concentration reached 28%. Then 0.6% (based on the starting material) of caustic soda, 2.2% (based on the starting material) of sodium silicate No. 3, 3.5% (based an the starting material) of 30% hydrogen peroxide and 0.2% (based on the starting material) of each of the deinking agents listed in Table 61 were added thereto. After adjusting the pulp concentration to 22% with water, the slurry was mixed in the bench disintegrator for 1 minute and then kneaded with a biaxial laboratory kneader at 300 rpm. After diluting with water so as to give a pulp concentration of 4%, it was disintegrated again with the bench disintegrator for 30 seconds. The obtained slurry was diluted with water so as to give a pulp concentration of 1% and then subjected to flotation at 30°C for 10 minutes. During the flotation process, 0.5% (based on the starting material) of CaCl<sub>2</sub> was added thereto. After the completion of the flotation, the pulp slurry was concentrated with an 80-mesh wire to thereby give a concentration of 4%. Then it was diluted with water so as to give a pulp concentration of 1% and treated with a TAPPI standard sheet machine to thereby give a pulp sheet.

The hardness of the employed water was adjusted to 10° dH with the use of CaCl<sub>2</sub> and MgCl<sub>2</sub> (Ca/Mg = 8/2 by mol).

The whiteness and b value of the pulp sheet thus obtained was measured with a color difference meter and the unliberated ink spots contained therein were counted with an image analyzer (100 x magnification).

Table 61 shows the component (b), the composition of the component (a) and the weight ratio of the components (b) to (a) of each of the tested deinking agents, while Table 62 shows the deinking performance thereof.

The components (b) listed in Table 61 were cod, sardine, saury and mackerel oil fatty acids (No. 74 - No. 77; T, U, V and W) and the one of No. 78 (X) was a mixture of the components (b) of No. 74 (T) and No. 75 (U) at a weight ratio of 50/50. Those of No. 79 to No. 80 (Y and Z) were prepared by varying the iodine value (IV) of the one of No. 75 (T).

Table 61

	Γ	1						
Definition and		Compenent (0)		component (8)				1.11.11
. Po		fatty acid	netural fat	polyhydric alcohol	g/p	alkylene oxide	de	(Mt. ratio)
	7			ren	(moter ratto)	type, molar ratio mol no.	mol no.	
-	7	•	hardened beef tailow (IV - 2.0) glycerol	glycerol .	1/0.6	80/80(1.8/1.8)	9:10	
prodects	۶	Đ	coconut oil	Gellot riose	1/0.3	E0/P0(2.0/1.0)	121.2	32/26
	7	>	linseed oil	cellobiose	1/0.0	E0/P9(3.3/1.9)	:	
	£	2	hardened (ish oil (IV = 0.8)	athylane glycol	1/1.6	EO/P013.9/1.61		
	7	×	soybean oil .	penteerythritol	1/1.2	E0/P0(6:2/1.0)	113.5	
	=		palm oil	D-glycero-D-qalactcheptose	1/0.7	E0/P0 (0. 4/1.0)	197.5	61/10
	8	,	rapeseed oil	propylene glycol	1/2.1	EO/PO(0.2/1.0)	36	52/46

#### Table 62

Deinking age	ent No.	Whiteness (%)	b value (%)	Unliberated ink no.
Invention	74	58.5	10.8	1
products	75	56.6	10.9	1 .
	76	56.7	10.9	1
	77	56.5	10.7	1
	78	56.6	10.9	. 0
	79	56.7	10.9	1
	80	56.7	10.7	1

#### Claims

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- 1. A deinking agent which
  - 1) comprises a mixture (b) of higher fatty acids having 8 to 24 carbon atoms, or salts thereof, wherein the average carbon atom number of said fatty acids in said mixture ranges from 12.7 to 22.5, and wherein the content of higher fatty acids having 20 to 24 carbon atoms or salts thereof, ranges from 9.6 to 70.6% by weight, said mixture having an iodine value (IV) of not greater than 45, and
  - 2) does not include alkylbenzenesulfonates.
  - 2. A deinking agent as claimed in claim 1, which furthermore comprises at least one surfactant (a) selected from the group consisting of compounds represented by the following general formulae (a-1), (a-2), (a-3) and (a-4) which follows:

$$R_1-O-(-AO-)_p-H$$
 (a-1)

wherein R<sub>1</sub> represents an alkyl or alkenyl group having 8 to 24 carbon atoms or an alkylphenyl group wherein said alkyl group has 6 to 14 carbon atoms,

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain wherein two or more kinds of the alkylene oxide are present; and p is 1 or above so as to result in a total molecular weight of from 800 to 10,000;

wherein R2 represents an alkyl or alkenyl group having 7 to 23 carbon atoms,

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain wherein two or more types of the alkylene oxide are present; and m is 1 or above so as to result in a total molecular weight of from 800 to 10,000;

$$R_3$$
-O-(-AO-)<sub>n</sub>-SO<sub>3</sub>M (a-3)

wherein R<sub>3</sub> represents an alkyl, alkenyl or cycloalkyl group having 8 to 24 carbon atoms or an alkylphenyl group wherein said alkyl has from 8 to 12 carbon atoms,

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain wherein two or more types of the alkylene oxide are present;

n is from 1 to 5; and

(a-4) a reaction product of an alkylene oxide with a mixture of a natural fat and polyhydric alcohol; and

M represents an alkali metal or ammonium ion.

3. A deinking agent as claimed in claim 2, wherein the weight ratio of each of the components (a-1) to (a-

4) to the component (b) falls within the following range:

(a-1)/(b) = 10/90 - 90/10;(a-2)/(b) = 10/90 - 90/10;

(a-3)/(b) = 5/95 - 30/70; and

(a-4)/(b) = 10/90 - 70/30.

- 4. A deinking agent as claimed in any of the preceding claims which contains 2.0 to 33.2% by weight of a fatty acid having 20 carbon atoms or a salt thereof and 9.5 to 32.0% by weight of a fatty acid having 22 carbon atoms.
  - 5. A deinking agent as claimed in any of the preceding claims wherein the higher fatty acids having 8 to 24 carbon atoms or salts thereof are semi-hardened or hardened fish oil fatty acids or salts thereof.
- 6. A method for deinking printed waste paper by the flotation process, the washing process, or a combination of said flotation or washing process, wherein the deinking agent as claimed in any of the claims 1-5 is added in the process.
- 7. A deinking method for reclaiming ink-containing waste paper having a pulping stage and a mixing or flotation stage which comprises:
  - (a) adding during said pulping stage, a deinking agent which 1) comprises a mixture of higher fatty acids having 8 to 24 carbon atoms, or salts thereof, wherein the average carbon atom number of said fatty acids in said mixture ranges from 12.7 to 21.5, and wherein the content of higher fatty acids having 20 to 24 carbon atoms or salts thereof, ranges from 9.6 to 70.6% by weight, said mixture having an iodine value (IV) of not greater than 45, and 2) does not include alkylbenzenesulfonates; and
  - (b) adding during said mixing or flotation stage, at least one surfactant selected from the group consisting of compounds represented by the following general formulae (a-1), (a-2), (a-3) and (a-4) which follow:

 $R_1$ -O-(-AO-)<sub>D</sub>-H (a-1)

wherein R<sub>1</sub> represents an alkyl or alkenyl group having 8 to 24 carbon atoms or an alkylphenyl group wherein said alkyl group has 6 to 14 carbon atoms;

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain wherein two or more kinds of the alkylene oxide are present; and p is 1 or above so as to result in a total molecular weight of from 800 to 10,000;

 $R_2$ -COO-(-AO-)<sub>m</sub>-H (a-2)

wherein R2 represents an alkyl or alkenyl group having 7 to 23 carbon atoms;

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain wherein two or more types of the alkylene oxide are present; and m is 1 or above so as to result in a total molecular weight of from 800 to 10,000; and

 $R_3$ -0-(-AO-)<sub>n</sub>-SO<sub>3</sub> M (a-3)

wherein R<sub>3</sub> represents an alkyl, alkenyl or cycloalkyl group having 8 to 24 carbon atoms or an alkylphenyl group wherein said alkyl has from 8 to 12 carbon atoms;

AO represents an alkylene oxide having 2 to 4 carbon atoms which may be either a block polymer chain or a random polymer chain wherein two or more types of the alkylene oxide are present; n is from 1 to 5; and

(a-4) a reaction product of an alkylene oxide with a mixture of a natural fat and polyhydric alcohol; and

M represents an alkali metal or ammonium ion.

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Category	Citation of document with ind of relevant pass	lication, where appropriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. CL5)
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